

**DEMONSTRATION RESULTS
OF
HOT GAS DECONTAMINATION
FOR EXPLOSIVES**

at

HAWTHORNE ARMY DEPOT

Hawthorne Nevada 89415-0015

VOLUME I OF IV

FINAL

Prepared for

U.S. ARMY ENVIRONMENTAL CENTER

Aberdeen Proving Ground, Maryland 21010-5401

Prepared by

THE TENNESSEE VALLEY AUTHORITY

ENVIRONMENTAL RESEARCH CENTER

Muscle Shoals, Alabama 35660-1010

SEPTEMBER 1995

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION / AVAILABILITY OF REPORT		
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE			Unlimited		
4. PERFORMING ORGANIZATION REPORT NUMBER(S)			5. MONITORING ORGANIZATION REPORT NUMBER(S)		
			SFIM-AEC-ET-CR-95031		
6a. NAME OF PERFORMING ORGANIZATION Tennessee Valley Authority		6b. OFFICE SYMBOL (If applicable) CEB 4C		7a. NAME OF MONITORING ORGANIZATION U.S. Army Environmental Center	
6c. ADDRESS (City, State, and ZIP Code) TVA Reservation Post Office Box 1010 Muscle Shoals, Alabama 35660-1010			7b. ADDRESS (City, State, and ZIP Code) Aberdeen Proving Grounds, Maryland 21010-5401		
8a. NAME OF FUNDING / SPONSORING ORGANIZATION U.S. Army Environmental Center		8b. OFFICE SYMBOL (If applicable) SFIM-AEC-TSD		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER TVA Contract No. TV-87179	
8c. ADDRESS (City, State, and ZIP Code) Aberdeen Proving Grounds, Maryland 21010-5401			10. SOURCE OF FUNDING NUMBERS		
			PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.
			WORK UNIT ACCESSION NO.		
11. TITLE (Include Security Classification) Demonstration Results of Hot Gas Decontamination for Explosives at Hawthorne Army Depot					
12. PERSONAL AUTHOR(S) T. R. Stumpe, J. R. Watson, M. C. Crim, C. W. Brown, Jr., J. J. Hoagland, W. J. Rogers					
13a. TYPE OF REPORT Final		13b. TIME COVERED FROM _____ TO _____		14. DATE OF REPORT (Year, Month, Day) 1995, September	
15. PAGE COUNT					
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP			
19. ABSTRACT (Continue on reverse if necessary and identify by block number) TVA demonstrated at Hawthorne Army Depot in June-October 1994 that the Hot Gas Decontamination (HGD) process was effective in removing and destroying residues of explosives from obsolete munitions as the final step of the demilitarization process. Prior testing demonstrated the applicability of the process to decontaminate residues of TNT and Ammonium Picrate from various items singly or in small lots. The present tests extended the process to additional munitions and explosives and tested them in quantities approaching the projected usage of the facility in routine operations. Specimens of actual contaminated munitions were examined in a baseline study to determine whether spiking levels were representative of actual conditions and whether sampling and analytical methods prescribed for spiked munitions were effective for actual contaminants and residues. The test program was comprised of 34 tests of five explosives (Ammonium Picrate, Comp A-3; Comp B, HBX, and TNT) and six munitions (3-inch, 5-inch, 106mm, and 175mm projectiles, MK 25 Ship Mines, and MK 54 Depth Bombs) with treatment times of 6 to 32 hours					
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL W. J. Hoagland			22b. TELEPHONE (Include Area Code) 410-612-6858		22c. OFFICE SYMBOL SFIM-AEC-ETD

ABBREVIATIONS

106mm	- U.S. Army, 106 millimeter artillery projectile
175mm	- U.S. Army, 175 millimeter artillery projectile
3"/5"	- U.S. Navy Projectile (also listed as 3-Inch/5-Inch)
ALSS	- Analytical Laboratory Support Services (TVA, Resource Group at Muscle Shoals, Alabama)
CEM	- Continuous Emissions Monitor
cm	- Centimeter
CO	- Carbon Monoxide
CO ₂	- Carbon Dioxide
Comp A-3	- Explosive made from RDX and Wax
Comp B	- Explosive made from TNT, RDX, and Wax
CRREL	- Cold Regions Research and Engineering Laboratory
DNB	- Dinitrobenzene
DNT	- Dinitrotoluene
DRE	- Destruction and Removal Efficiency
DZB	- Day and Zimmerman/Basil Corporation, operating contractor at HWAD
EPA	- Environmental Protection Agency
FF-13	- Flash Furnace Facility at HWAD
g	- gram
GLP-0001	- TVA/ALSS Laboratory Procedure Preparation
GLP-0003	- TVA/ALSS Procedure Preparation and Distribution
GLP-0005	- TVA/ALSS Nonconformances and Corrective Actions
GLP-0007	- TVA/ALSS Control of Changes to Software
H&S	- Health and Safety
HBX	- Explosive made from TNT, RDX, Aluminum, Lecithin, and Wax
HGD	- Hot Gas Decontamination
HMX	- Octrahydro - 1, 3, 5, 7 - tertranitro -1, 3, 5, 7 - tertrazocine
HPLC	- High Performance Liquid Chromatography
HWAD	- Hawthorne Army Depot, Hawthorne, Nevada
ID	- Induced Draft
LCD	- Liquid Crystal Display
lb	- Pounds
ma	- Milliamp
MDL	- Method Detection Limit
MK 25	- U.S. Navy Ship Mine
MK 54	- U.S. Navy Depth Bomb
mL	- Milliliter
mm	- Millimeter
MM5	- Modified Method 5 - EPA sampling procedure
MSDS	- Material Safety Data Sheet
MX	- Matrix Spike or Matrix Spike Duplicate Recovery was outside limits due to suspected matrix effects
NA	- Compound Not Analyzed; Not Applicable
NAD	- Naval Ammunition Depot
NB	- Nitrobenzene
NC	- Not Calculable
ND	- Compound Not Detected
NO _x	- Nitrogen Oxides
NR	- No Record
O&M	- Operating and Maintenance
OB-OD	- Open Burning-Open Detonation

ABBREVIATIONS (Continued)

ppm	- Parts per Million
QA/QC	- Quality Assurance/Quality Control
RCRA	- Resource Conservation and Recovery Act
RDX	- Hexahydro - 1, 3, 5 - trinitro - 1, 3, 5 - triazine
RFW	- Roy F. Weston
SDEV	- Standard Deviation
SARM	- Standard Analytical Reference Material
SD	- Surrogate Recovery Low Due to Dilution
SO ₂	- Sulfur Dioxide
SO _x	- Sulfur Oxides
SM	- Surrogate Recovery Out of Limits, Matrix Effect Suspected
SP-001	- TVA/ALSS Sample Chain of Custody Procedure
sq cm	- Square Centimeter
THC	- Total Hydrocarbons
TNB	- Trinitrobenzene
TNT	- 2, 4, 6 - Trinitrotoluene
TR	- Compound Present At Trace Level
TVA	- The Tennessee Valley Authority
USACHPPM	- U.S. Army Center for Health Promotion and Preventive Medicine
USADACS	- U.S. Army Defense Ammunition Center and School
USAEC	- U.S. Army Environmental Center
WADF	- Western Area Demilitarization Facility
Yellow D	- Ammonium Picrate
°C	- Degrees Celsius
°F	- Degrees Fahrenheit
µg	- Microgram

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					Temperature Data			CEM Data			Sampling Data			
					15 min.	15 sec	1 min.	1 min.	1 min.	1 min.	Test Items	Chamber	Acc. Chambr	(Tables)
A	N/A		D-9											
B	22 Jun	175mm Projectiles	D-10	4-12	F-1							F-2		F-1
1	26 Jun	3-inch Projectiles	D-11	4-7										
3	28 Jun	175mm Projectiles	D-12	4-2										F-3
B2	30 Jun	Empty car only	D-13											F-4
B3	3 Jul	None	D-14									F-5		
B4	4 Jul	None	D-15									F-6		
5	10 Jul	3-inch Projectiles	D-16	4-4								F-7		
2	12 Jul	3-inch/5-inch Projectile	D-17	4-6	F-2			F-3			F-8	F-9		
4	15 Jul	3-inch Projectiles	D-18	4-7	F-4			F-5			F-10	F-11		
6	16 Jul	MK 25 Ship Mines	D-19	4-8	F-6			F-7			F-12	F-13		
7	22 Jul	3-inch/5-inch Projectile	D-20	4-6	F-8			F-9			F-14	F-15		
8	24 Jul	175mm Projectiles	D-21	4-2	F-10			F-11			F-16	F-17		
9	26 Jul	3-inch Projectiles	D-22	4-4	F-12			F-13			F-18	F-19		F-79
10	29 Jul	3-inch/5-inch Projectile	D-23	4-10	F-14			F-15			F-20	F-21		
1-10			D-24		F-16			F-17			F-22	F-23		
C	1 Aug	None	D-25										F-24	
11	9 Aug	MK 25 Ship Mines	D-26	4-8								F-25		
12	13 Aug	3-inch/5-inch Projectile	D-27	4-6	F-18			F-19			F-26	F-27		
13	15 Aug	175mm Projectiles	D-28	4-2	F-20			F-21			F-28	F-29		F-80
14	17 Aug	3-inch Projectiles	D-29	4-4	F-22			F-23			F-30	F-31		
15	20 Aug	3-inch/5-inch Projectile	D-30	4-10	F-24			F-25			F-32	F-33		
16	21 Aug	MK 54 Depth Bombs	D-31	4-5	F-26			F-27			F-34	F-35		
17	25 Aug	3-inch/5-inch Projectile	D-32	4-6	F-28			F-29, F-30			F-36	F-37		
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Test #	Test Date 1994	Test Item	App. D Test Config.	Test Photo Number	Appendix F										
					Temperature Data		CEM Data		Sampling Data				(Tables)		
					15 min.	15 sec	15 min.	1 min.	Test Items	Chamber	Acc. Chamber	Insul.	Blank		
D	6 Sept	175mm Projectiles	D-37	4-13	F-41										
21	12 Sept	MK 54 Depth Bombs	D-38	4-5	F-42			F-43, F-44	F-48	F-49					F-47
22	16 Sept	106mm Projectiles	D-39	4-1	F-45			F-46	F-50	F-51					
23	18 Sept	175mm Projectiles	D-40	4-2	F-47			F-48	F-52	F-53					
24	20 Sept	3-inch/5-inch Projectiles	D-41	4-10	F-49			F-50	F-54	F-55				F-82	
25	21 Sept	MK 54 Depth Bombs	D-42	4-5	F-51			F-52, F-53	F-56	F-57					
26	25 Sept	MK 25 Ship Mines	D-43	4-9	F-54, F-58	F-55	F-56	F-57, F-59	F-58	F-59					
28	29 Sept	106mm Projectiles	D-44	4-1	F-60		F-61		F-60	F-61					
27	1 Oct	175mm Projectiles	D-45		F-62	F-63	F-64	F-65	F-62	F-63				F-83	
29	2 Oct	3-inch/5-inch Projectile	D-46	4-10	F-66	F-67	F-68	F-69	F-64	F-65					
1-29			D-47									F-66			
"96"	13 Oct	175mm Projectiles	D-48	4-11	F-70	F-71	F-72	F-73	F-67						
30	15 Oct	175mm Projectiles	D-49, 49a	4-3	F-74	F-75	F-76	F-77	F-68	F-69					
31	19 Oct	175mm Projectiles	D-50, 50a	4-3	F-78	F-79	F-80	F-81	F-70	F-71					
32	23 Oct	175mm Projectiles	D-51, 51a	4-3	F-82	F-83	F-84	F-85	F-72	F-73					
33	26 Oct	175mm Projectiles	D-52, 52a	4-3	F-86	F-87	F-88	F-89	F-74	F-75					
34	29 Oct	175mm Projectiles	D-53, 53a	4-3	F-90	F-91	F-92	F-93	F-76	F-77				F-84	
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1.0 SUMMARY

During June-October, 1994, the Tennessee Valley Authority (TVA) successfully demonstrated at Hawthorne Army Depot (HWAD) that the Hot Gas Decontamination (HGD) process is safe, efficient, and economical in removing and destroying residues of explosives from obsolete munitions as the final step of the demilitarization process. Prior testing performed by Roy F. Weston, Inc. demonstrated the applicability of the process to decontaminate TNT and Ammonium Picrate residues from small lots of obsolete munitions, pipes, and equipment. The current demonstration program refined the operational parameters and ability to decontaminate additional explosives and munitions in quantities up to the production capacity of the facility.

The concept of HGD is that a flow of hot gas (in the case of HWAD, it is comprised of heated air and flue gases from the thermal oxidizer exhaust) at a temperature as low as 550°F (288°C), can sublime and/or thermally degrade organic contaminants such as residual explosives without causing them to explode. The vapors of the explosive compounds are then directed to a thermal oxidizer unit where they are destroyed under controlled conditions. Only combustion products exited the system. The explosive vapors are mineralized, that is, they are converted to low molecular weight inorganic compounds, predominantly carbon dioxide and water. A small amount of oxides of nitrogen (NO_x) is produced from the nitrogen content of the explosives themselves but oxidizer conditions are controlled to minimize NO_x formation from combustion air. Oxides of sulfur (SO_x) are produced from the sulfur content of the diesel fuel used to fire the thermal oxidizer. Modern explosives do not contain sulfur or chlorine compounds to contribute to emissions.

The demonstration program was comprised of 34 tests of five explosives (Ammonium Picrate, Comp A-3, Comp B, HBX, and TNT) and six munitions (3-inch, 5-inch, 106mm, and 175mm projectiles, MK 25 Ship Mines, and MK 54 Depth Bombs) with treatment times of 6 to 32 hours and treatment temperatures of 550 to 700°F (288 to 371°C). The analytical results indicate that HGD is effective in decontaminating munitions to quantities below the detection limits of the HPLC,

with destruction removal efficiencies (DRE) greater than 99.999 percent. The analytical values for 68.8 percent of the test items sampled/analyzed were less than the HPLC Method detection limits (MDL) of 0.007 µg/mL for RDX, 0.004 µg/mL for TNT, and 0.004 µg/mL for Ammonium Picrate. Furthermore, 30.3 percent of the test items had detectable microgram quantities of explosive residues with analytical results which gave DREs greater than 99.999%, and the remaining 1.1% of the test items had DREs of 99.979 to 99.998%. Based on this extensive demonstration program, it was proven that the HGD process is safe, efficient, and economical in decontaminating explosives residue contaminated munitions.

To determine if any explosive vapors were being deposited and accumulated on interior surfaces of the system 8 wipe samples were taken at various locations within the system such as chamber liner chamber floor, ductwork, and I.D. fan blades, after each individual demonstration test, after the completion of each series of 10 demonstration tests, and after the final demonstration test. The analytical results indicated that microgram quantities of explosive residue were randomly detected and that no accumulation was taking place. The fact that only very small quantities (µg/cm²) of explosives were found in the chamber suggested that there was no buildup of explosives in the system. This was also supported by the fact that wipe samples were taken from different locations to determine the buildup rate between several tests. Since no accumulation was observed, it was determined that the system operation did not and would not present an explosive hazard to personnel and the facility during routine operation.

Atmospheric emissions sampling was performed by (U.S. Army Center for Health Promotion and Preventive Medicine (USACHPPM) on the thermal oxidizer exhaust stack during the final series of production capacity demonstration tests using melted out 175mm projectiles containing Comp B explosive residue. The results indicated that the emissions from the facility (total hydrocarbons, SO₂, NO_x, CO, particulates, and metals) were significantly below the Nevada allowable limits. On March 7, 1995, the Nevada Department of Environmental Protection granted permission for HWAD to begin production operation of their HGD facility based on

the results presented to them from this demonstration program and the emissions sampling events.

The total operating and maintenance (O&M) costs for the Hawthorne HGD facility, based on actual operating experience from this demonstration program is \$55.27 per short ton (\$60.87/metric ton) of metal processed which compares very favorably to the O&M cost of \$109.73 per short ton (\$120.85/metric ton) for operating HWAD's flashing furnace in Building 117-3 for 175mm projectiles. The HGD O&M costs includes operations and maintenance loaded labor costs, consumable costs (fuel, electricity, maintenance materials and miscellaneous costs), and sampling and analysis costs. This HGD O&M cost is also in the same range that was estimated for the new production HGD facility design (\$50.82 short ton, \$55.97 metric ton). (Reference #5, TVA Report). However, in comparison, based on processing 175mm projectiles during a 5-day, 40-hour week, the HWAD HGD facility can process approximately 83 short tons (75 metric tons) per week while it is estimated that the new HGD production facility can process 193 short tons (175 metric tons) per week.

2.0 INTRODUCTION

2.1 Concept

The concept of the Hot Gas Decontamination Process (HGD) is that a flow of hot gas, at a temperature as low as 550°F (288°C), can volatilize and/or thermally degrade organic contaminants such as residual explosives without causing them to explode. The vapors of the explosive compounds can then be directed to a thermal oxidation unit where they will be destroyed under controlled conditions. Only combustion products will exit the system. The explosives are mineralized, that is, they are converted to low molecular weight inorganic compounds, predominantly carbon dioxide and water. A small amount of oxides of nitrogen (NO_x) is produced from the nitrogen content of the explosives themselves but oxidizer conditions are controlled to minimize NO_x formation from combustion air. Modern explosives do not contain sulfur or chlorine compounds to contribute to emissions.

2.2 Background

2.2.1 Process Description

For each test, explosive-contaminated munitions were loaded into the chamber and the chamber secured. The thermal oxidizer was brought up to an operating temperature of about 1800°F (982°C). The thermal oxidizer was maintained at operating temperature throughout the chamber heatup, steady state, and cooldown periods of the test. Hot gas was then recirculated from the exhaust stack through the chamber to volatilize the explosives from the test munitions. The hot gas was ducted into the chamber and distributed through gas diffusers. The contaminated exhaust gas was pulled from the chamber and directed to the thermal oxidizer by an induced-draft I.D. fan. The system was maintained under negative pressure by the fan to prevent release of gases to the environment. The organic contaminants in the chamber exhaust gases were destroyed in the thermal oxidizer before discharge to the atmosphere. Following the

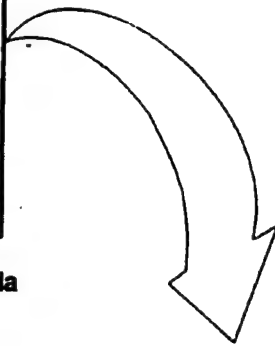
cooldown period, the treated test munitions were removed from the chamber and sampled. Operation of the system was monitored by an instrumentation and control system located in Building 117-3. Temperature thermocouples located inside the munitions provided input to a data acquisition system located in Building 117-15. Personnel monitored munition temperatures during the test from this location. The control system contained safety interlocks that automatically shutdown the system in the event of an upset condition such as a positive pressure in the chamber.

2.2.2 Site Description

Figure 2-1 is a map of the west-central portion of the state of Nevada showing the location of Hawthorne. Hawthorne Army Depot (HWAD) covers some 148,000 acres of land situated in Mineral County approximately 135 miles southeast of Reno, Nevada, along U.S. Route 95. HWAD was originally constructed in 1929 as a U.S. Navy Ammunition Depot (NAD). The Western Area Demilitarization Facility (WADF) was constructed by the Navy from 1975 to 1977 to dispose of Naval munitions in a controlled manner (Reference #1). The U.S. Army continued the Navy's munitions demilitarization program and assumed control of Hawthorne in October 1977 and changed the name to Hawthorne Army Ammunition Plant. It was reclassified as a depot and the name changed to Hawthorne Army Depot (HWAD) in 1994.

The WADF, on the northern end of the installation boundary near Walker Lake, is shown in Figure 2-2.

The WADF conducts a process of reverse assembly of the munitions followed by steam, autoclave, mechanical, or washout removal of explosive charges. Reclaimed explosives can be recycled, or employed in less demanding applications such as demolitions or as donor explosives for



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safe disposal of unexploded ordinance. Small metal parts are decontaminated by furnace flashing. Large items could be decontaminated in Building 117-15. This chamber was originally designed and constructed to provide for flashing large items. The parts are then released as scrap metal. A detailed view of the WADF is shown in Figure 2-3.

An enclosed chamber was constructed at WADF for the purpose of decontaminating items by flashing them with surplus propellant powder. This approach was not successful and the chamber has been adapted to the HGD which has been demonstrated to decontaminate individual items under test conditions (Reference #2 RFW Report). Figure 2-4 shows the area of the flashing furnace and other support buildings used for this test plan.

The primary buildings used during the demonstration program are as follows:

- Building 117-1 (Service and Support) - This building contains the laboratory and was used by TVA personnel for sample preparations and some analytical work. (The detailed analyses of each sample was conducted at TVA laboratories in Muscle Shoals, Alabama.) This building also contains shower facilities for all personnel.
- Building 117-3 (Decontamination and Small Items Building) - Operations support area for TVA personnel. This building contains the control room for the HGD system. Break room, restrooms, and lunch areas are also in this building.

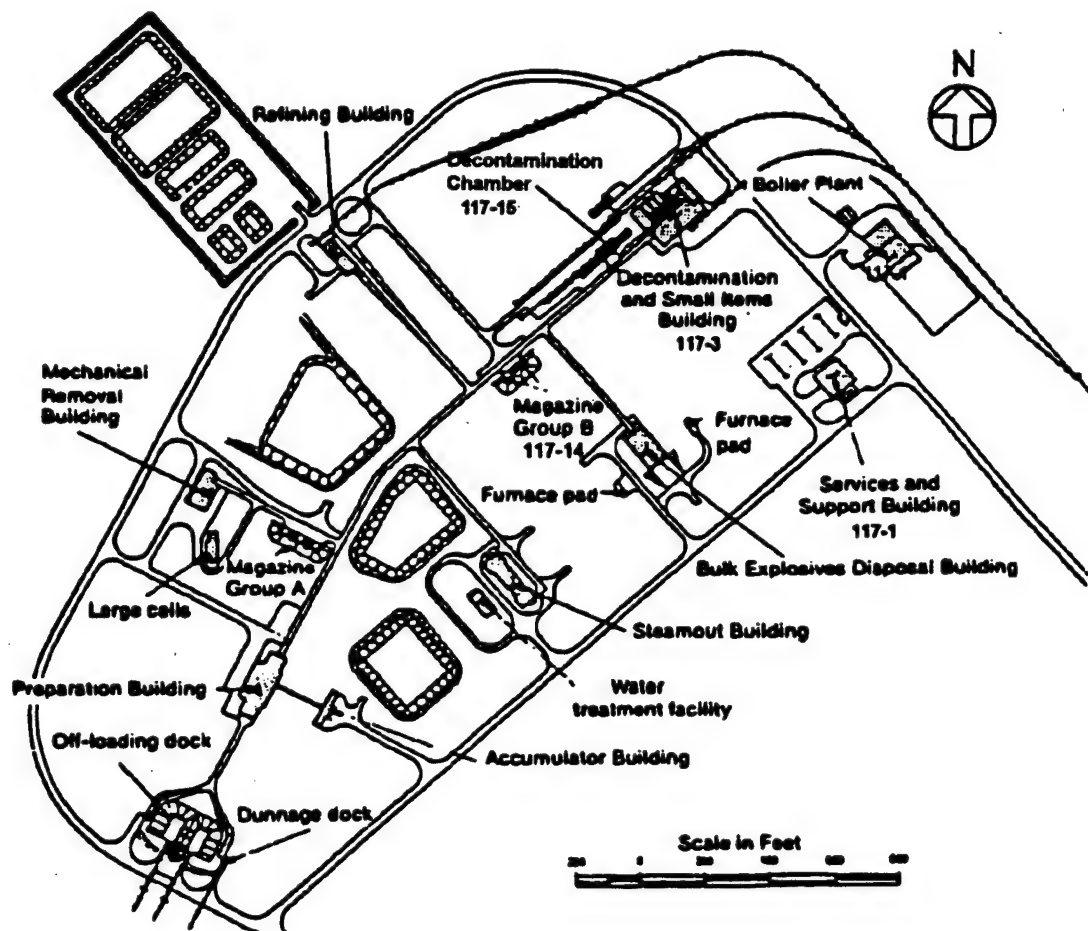


Figure 2-3 Overall View of the Western Area Demilitarization Facility

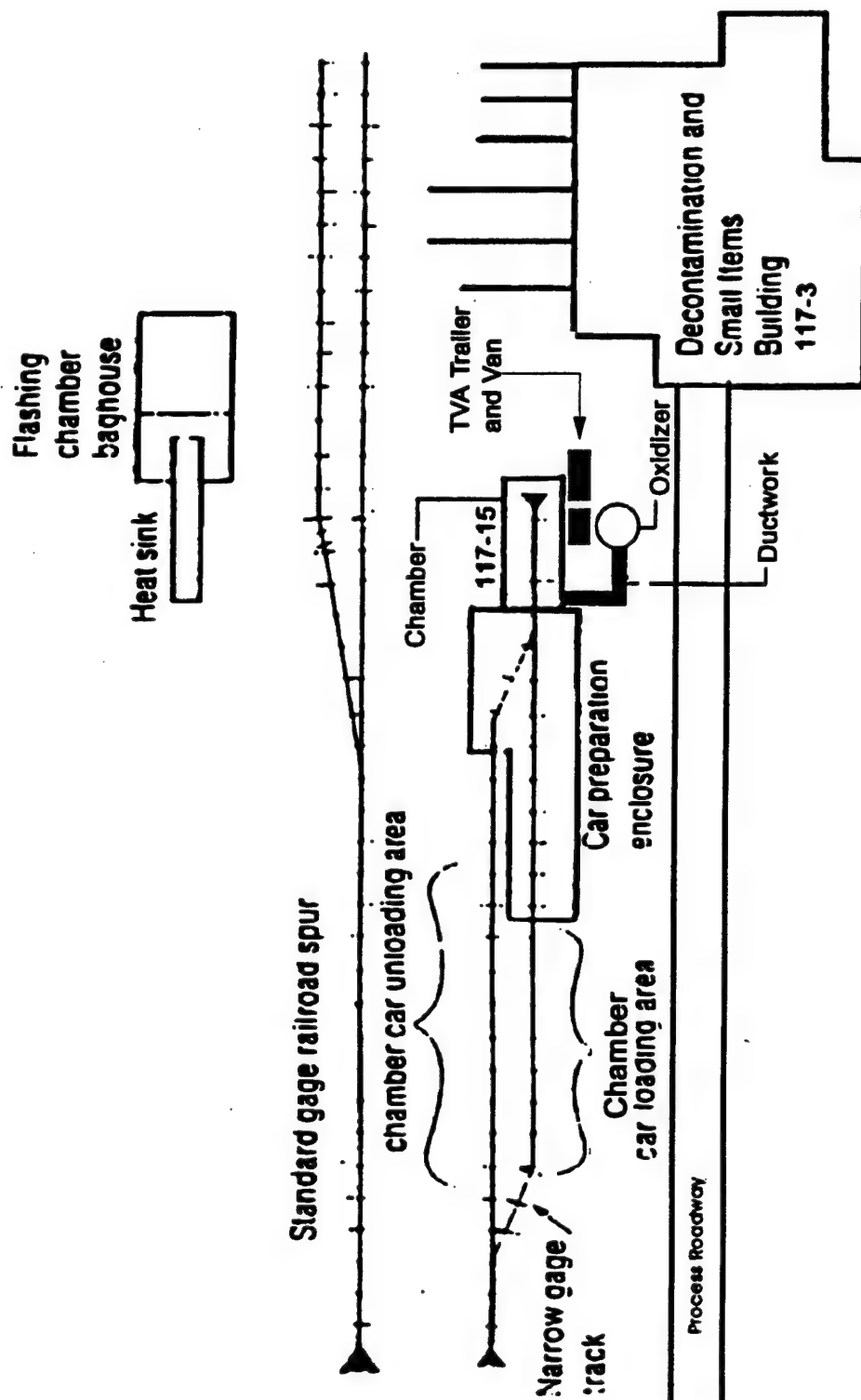


Figure 2-4 Arrangement of the Chamber (117-15) and Small Items Building (117-3)

- **Building 117-14 (Explosive Storage Bunker at Magazine Group B) -** This facility was used to store, weigh, and package predetermined sample size quantities of explosive material for explosive spiking. Explosives prepared for samples can be stored at this facility in nonstandard pack. Quantity of material did not exceed 20 pounds net explosive weight.
- **Building 117-15 (Decontamination Chamber) -** The testing of explosives-contaminated munitions, ship mines, and depth bombs was performed inside this facility. The chamber car loading area and car preparation enclosure were used to configure each flat car for the appropriate test. TVA located the control and sample van near this building (Photos E-1 and E-2).

2.2.3 Initial Tests

The process was originally tested as a means for decontaminating the structure of a building in a munitions plant. Hot gas was ducted from a preheater through the building to volatilize the explosives deposited on the materials of construction (predominantly concrete block) over years of plant operation. The gas and volatilized explosives then passed out of the building to the oxidizer as described above. This initial test was conducted at Cornhusker Army Ammunition Plant near Grand Island, Nebraska, by A. D. Little Inc. in 1987. (Reference #4, AD Little Report.)

After that test was satisfactorily concluded, the preheater and oxidizer were transferred to HWAD in 1988 and installed on the WADF flash chamber. The chamber was otherwise modified to apply the HGD process, by the installation of ducting, instrumentation, and controls. A false wall was installed across the chamber to reduce the working length of the chamber. Currently, the chamber can accommodate only one of the narrow gauge railcars provided to carry items for treatment.

2.2.4 Weston Tests

To determine the feasibility of the hot gas decontamination of explosive-contaminated equipment, a pilot study was conducted from July 10 to September 21, 1989, by Roy F. Weston, Inc. (Reference #2 RFW report.) Nine tests were conducted on test items contaminated with TNT; one test was conducted on Ammonium Picrate (Yellow D). For the tests evaluating TNT, the test duration was 6, 12, 24, or 36 hours (after reaching steady state) and test temperatures of 400°F (204°C), 500°F (260°C), or 600°F (316°C) were evaluated. A residence time of 48 hours and a temperature of 600°F (316°C) was used for the evaluation of Ammonium Picrate; these test conditions were selected to ensure the decontamination process would be complete and to avoid potential safety problems associated with partially decomposed Ammonium Picrate (picric acid). To determine the destruction and removal efficiency (DRE) of the process, stack testing was conducted during the first three TNT tests at the thermal oxidizer inlet and outlet.

The results of the pilot study indicate that a minimum temperature of 500°F (260°C) and a residence time of 6 hours are required to effectively decontaminate test items contaminated with TNT. Operating conditions of 600°F (316°C) and 48 hours are sufficient to treat items contaminated with Ammonium Picrate. Stack test results indicate that the DRE for TNT exceeds 99.99 percent.

However, these tests were made with one or a few items at a time and the sampling and analytical procedures employed were not fully adapted to the process and its conditions. Therefore, the test results, while encouraging, were not rigorous enough to assure decontamination comparable to the traditional flashing process.

2.2.5 Plant Modifications

The test report of the Weston tests contained several recommendations for improvement of the system. These recommendations were formulated into a statement of work and were funded in the first quarter of FY 92.

The plant operating contractor, Day and Zimmerman/Basil Corporation (DZB) was responsible for the modifications to the facility and installation of the process equipment. The modifications included:

- Conversion of the thermal oxidizer fuel supply from propane to diesel fuel because of the ready availability of diesel fuel at HWAD.
- Removal of the air preheater and combustion fan and replacement with a collection plate, recirculating duct, and recycle fan that diverted the hot oxidizer exhaust gas back to the chamber.
- Insulation of the chamber to minimize heat losses.
- Relocation of the system controls from the chamber facility (Building 117-15) to the control room at the support facility (Building 117-3) to return the operation back to a hazardous (explosion proof) electrical classification.

Modifications and equipment installation were completed on August 27, 1993.

2.2.6 DZB Proveout

To determine the satisfactory completion of the modifications to the facility three shakedown tests were conducted by DZB between August 31 and September 8, 1993 (Reference #3 DZB report). The test conditions for the three tests were as follows:

- Test #1 Raise the chamber temperature to 500°F (260°C) and maintain for 12 hours.
- Test #2 Raise the chamber temperature to 800°F (427°C) and maintain for 8 hours.
- Test #3 Raise the chamber temperature to 500°F (260°C) and maintain for 1 hour and simulate an emergency shutdown.

The objectives of all three tests were met. The overall conclusion from the results of the shakedown tests was that the system was operational and would be capable of unattended production operation. Although the chamber temperature reached 800°F (427°C) and sustained it for approximately 6 hours, inherent heat losses in the system keep this temperature from being a normal operating temperature. Chamber temperatures of up to 750°F (399°C) will be attainable with possibilities of achieving 800°F (427°C) by resolving heat loss problems.

2.2.7 Process Equipment

Figure 2-5 shows the process flow diagram with its major equipment items. The following is a brief summary of the principal components of the system:

Flash Chamber

The chamber (Photo E-3) is a 12.5 feet (3.8 meters) diameter, 1 inch (2.54 centimeter) thick cylindrical steel structure encased in 4 to 6 feet (1.2 to 1.8 meters) of reinforced concrete. Two inches (5 centimeters) of fiberglass insulation is located between the steel and the reinforced concrete. The four inches (10 centimeters) of calcium silicate insulation with a 10 gauge (2.59 millimeters) carbon steel jacket is installed on the inside surface of the chamber steel tube. A temporary false wall reducing the length of the 53 foot (16.2 meters) chamber to 30 feet (9.1 meters) is also provided to reduce heat requirements and losses. The false wall is constructed of ¼ inch (.635 centimeters) carbon steel plate with 6 inches

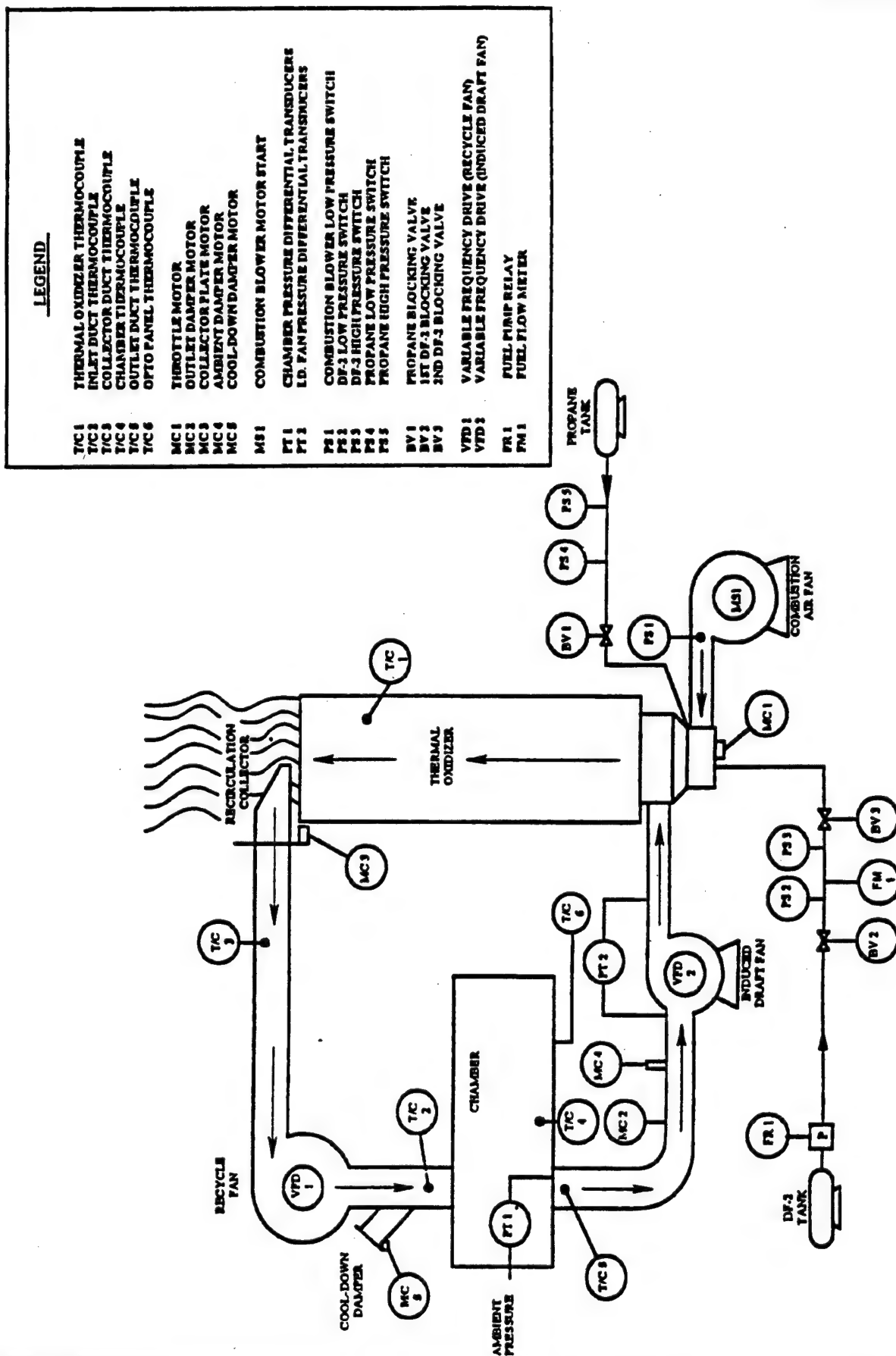


Figure 2-5 Hot Gas Decontamination Facility Flow and Control Diagram

(15 centimeters) of calcium silicate insulation. Entry to the chamber is provided by a motor driven 16 inch (41 centimeters) thick blast resistant door constructed of steel and concrete.

Hot gas is introduced to the rear of the chamber through ducting that is 18 inches (46 centimeters) in diameter. The inlet air ducts in the chamber were modified to accommodate this ducting. The modification involved cutting two 18-inch (46 centimeters) diameter holes through the reinforced concrete wall surrounding the chamber. Hot gas is distributed throughout the chamber by diffusers, as shown in Figure 2-6 and Photo E-4.

Thermal Oxidizer

A vertical thermal oxidizer, or afterburner, is used to destroy contaminated gases vented from the chamber. The oxidizer chamber is 5 feet (1.5 meters) in diameter and 20 feet (6.1 meters) tall. It is lined with 3 inches (8 centimeters) of ceramic fiber insulation. The unit is diesel-fired and rated for a total heat release of 4.5 million Btu/hour (1319 KW). The exhaust gases from the flash chamber can be heated in the thermal oxidizer to a maximum temperature of approximately 2,000°F (1093°C) depending upon ambient temperature and wind conditions. The oxidizer is preceded by an induced draft fan, which withdraws gases from the chamber and maintains a negative pressure on the system. The fan is rated at 2,200 scfm (1038 L/s) of gases. During the demonstration program, the oxidizer retention time was approximately 2.4 seconds based on an 1800°F (982°C) temperature and a gas flowrate of 2000 scfm (944 STD L/s).

System Controller

The control system used was a Maples 590D Controller serially interfaced with four Opto 22 I/O boards (2 analog and 2 digital). The Maples Controller is a microprocessor based process and machine controller. The

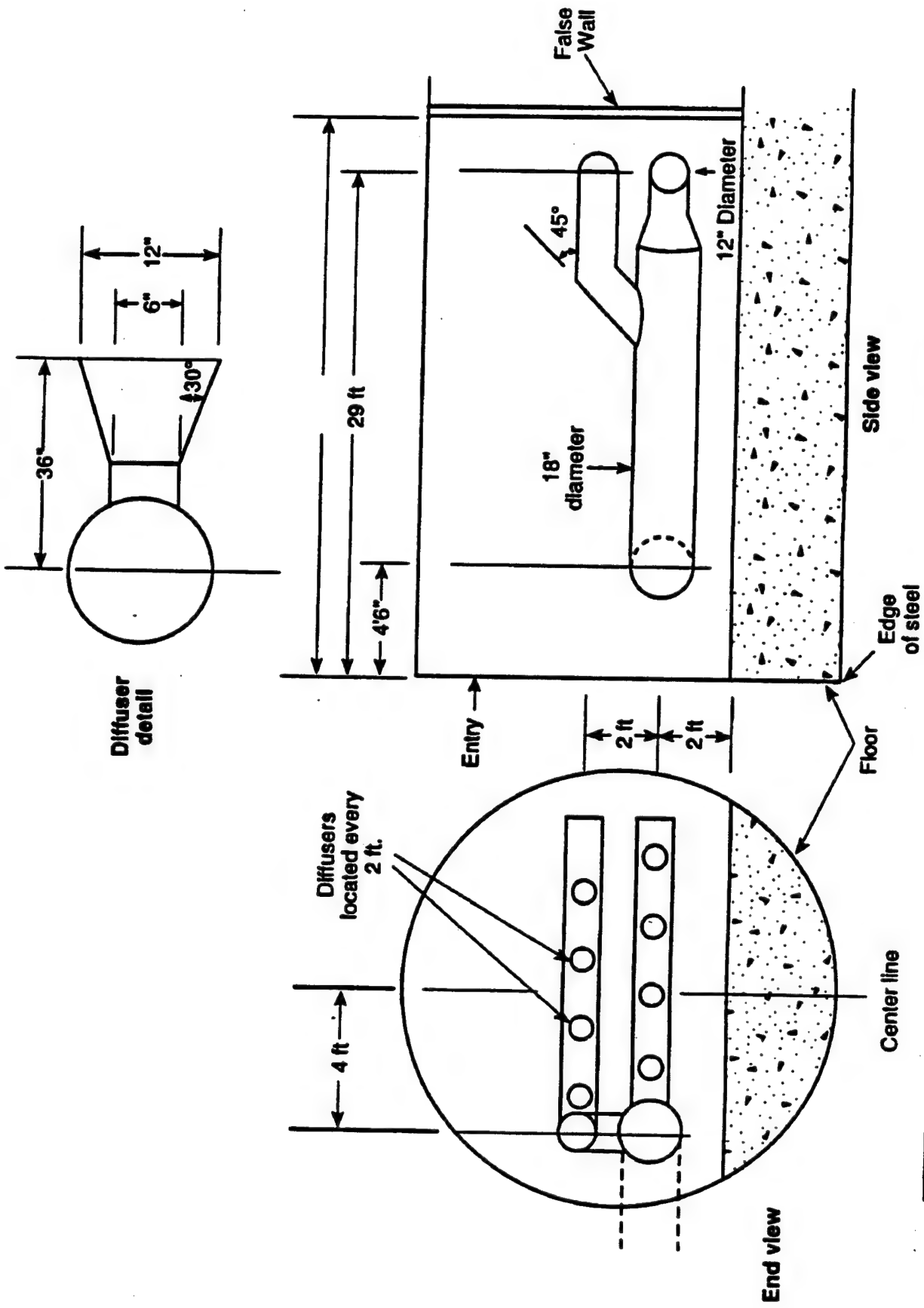


Figure 2-6 Schematic of Diffusers Used to Distribute Hot Gas in Chamber

controller contains sockets for user memory (RAM) and program memory (PROM), two asynchronous serial communication ports, one parallel I/O port (24 inputs and 24 outputs), 65 key keyboard and a 2x40 character Liquid Crystal Display (LCD). The software program used was written in Process Basic. The Opto 22 I/O boards communicate serially with the Maples controller using a serial data link. Two analog boards (one 8 position and one 16 position) and two digital boards (one 8 position and one 16 position) are used to communicate with variable speed drives, solenoid valves, thermocouples, limit switches and 4 20ma modules.

Data Logger

An Omega OM-372 Recorder/Data Logger is used to record internal temperatures of the munitions being tested. The temperature inputs to the data logger are from K-Type thermocouples with variable locations inside the chamber.

2.3 Project Objectives

The original scope of work included optimization of decontamination treatment conditions in order to conserve process time and energy usage. The objective of this test program de-emphasized optimization in favor of gaining the maximum confirmation and confidence in the efficacy of the decontamination process for the number of tests to be performed.

Optimization of the process and confirmation of the efficacy of the optimized treatment conditions would call for substantially more work. Experimental design would call for 11 tests to establish minimum operating costs based on process time and energy consumption. At least four additional tests per explosive (spiked munitions) would be needed to establish effective explosive decontamination. A minimum of five further tests per explosive (melted out munitions), all completed satisfactorily, would be considered necessary to confirm the efficacy of decontamination at those optimized conditions. That gives a total of 20 tests to yield

confirmed optimized conditions for one explosive. The program would therefore call for 100 tests to cover Comp A-3, Comp B, HBX, TNT, and Yellow D. This number of tests were not within the scope of this test program because of budget and time restraints.

2.3.1 Phase I - Tests at HWAD

The objective of Phase I of the HGD Demonstration Program was to develop data to demonstrate the efficacy and safety of the HGD process to remove and destroy residues of explosives from obsolete munitions as the final step of the demilitarization process. The demonstration program was comprised of preliminary studies of test items spiking (Photo E-5), sampling (Photo E-6), and analysis. Specimens of actual contaminated munitions items were examined in a baseline study to determine whether spiking levels used previously (in the Weston study, Reference #2) are representative of actual conditions, and whether sampling and analytical methods prescribed for artificially spiked items are effective for actual contaminants and residues. Spiking and sampling procedures were revised as necessary to better simulate expected contamination. The contaminated items and the corresponding explosives tested are shown in Table 2-1.

TABLE 2-1
CONTAMINATED ITEMS AND EXPLOSIVE COMPOUNDS TESTED

ITEM	EXPLOSIVE	CONDITION
1. 175mm Projectiles	Comp B	Spiked, Melted Out
2. 3-Inch/5-Inch Projectiles	RDX	Spiked
3. 106mm Projectiles	Comp A-3	Melted Out
4. 3-Inch Projectiles	HBX	Spiked
5. MK 54 Depth Bombs	HBX	Melted Out
6. 3-Inch Projectiles	TNT	Spiked
7. MK 25 Ship Mine	TNT	Spiked
8. 3-Inch/5-Inch Projectiles	Yellow D	Spiked

The demonstration program examined a number of variables to determine the efficacy of HGD in decontaminating the items of current interest. The major response variable was the effectiveness of explosives residue removal from the test items. It was based on the absence or presence of explosive residue after treatment, and, if present, its quantity. Other variables contributing to evaluation of the process included temperatures of the test items, the chamber, and other points in the system. Composition of gases discharged from the chamber and from the stack were variables that were monitored. Heatup and cooldown times for the system were noted. The major experimental control variables that were changed from test to test include the items treated, the type of contaminating explosive, placement of spiked or contaminated test items in the chamber, the nominal treatment temperature, and the residence time at that temperature. These variables constitute the basic description of the decontamination regimen; what is being treated to remove which contaminant, and under what conditions. Other variables were held constant throughout the program. They include all controllable aspects of the chamber operation besides the treatment time and temperature. All tests followed the same chamber operation with respect to air flow and chamber draft, heat delivery, and oxidizer operating parameters. Placement of the railcar with racks and total lading of items was held constant. Only the distribution of contaminated test items among inert items included to provide thermal mass was altered as an experimental control variable. Procedures for sampling and analysis of residues were standardized as described in this report, and results were evaluated objectively.

Some variables were independent and not subject to control or stabilization. It is likely that this category can be limited to ambient conditions of temperature and humidity, which might have some small effect on the temperature profile of the system.

2.3.1.1 Summary Test Plan

A series of 34-facility demonstration tests of the HGD process were conducted at WADF/HWAD. An additional seven operating cycles--two conducted with inert items to check temperature distribution; one to decontaminate an empty railcar; one performed as a preliminary test to a full chamber load test; and three to decontaminate the chamber were conducted. See the sequence of tests in Table 2-2. Samples were analyzed at TVA laboratories, with results returned to the field for ongoing process evaluation and test revision where necessary. Results from all the tests are presented in Appendix F.

The most desirable demonstration of the process would call for corresponding items and explosives to be treated in spiked condition for precise measurement and in demilitarized condition to demonstrate realistic treatment requirements. However, due to current availability of the types of decontaminated items to be spiked with explosives and of melted out items to be decontaminated from a realistic demilitarized condition, the table reflects some substitutions and adjustments.

There were large numbers of 175mm projectiles which had been furnace flashed in Building 117-5 (FF-13). Spiking tests of Comp B were conducted with these projectiles. Also, there was a large quantity of 175mm projectiles originally loaded with Comp B being demilitarized. These melted out projectiles were used for testing the decontamination of the residue of Comp B.

No solvent or miscible combination of solvents was identified which will produce a homogeneous solution of Comp A-3 suitable for spiking. Therefore, spike tests to represent Comp A-3 were made with neat RDX. This was considered an acceptable substitution by TVA and U.S. Army Environmental Center, Environmental Technology Division because RDX comprises 91 percent of Comp A-3, the remainder being a non-explosive

**TABLE 2-2
TEST SEQUENCE**

Test #	Test Date 1994	Test Item	# Items	Item Source	Test Status		Test Conditions		
					Explosive	Condition	Time	Temp. °F	Temp. °C
A	N/A								
B	22 Jun	175mm Projectiles	96	FF-13	None	None	6 / 24 Hrs	500 / 700	260 / 371
1	26 Jun	3-inch Projectiles	192	FF-13	TNT	Spiked	6 Hrs	500	260
3	28 Jun	175mm Projectiles	96	FF-13	Comp B	Spiked	6 Hrs	500	260
B2	30 Jun	Empty car only	N/A	N/A	N/A	N/A	6 Hrs	550	288
B3	3 Jul	None	N/A	N/A	N/A	N/A	6 Hrs	550	288
B4	4 Jul	None	N/A	N/A	N/A	N/A	24 Hrs	650	343
5	10 Jul	3-inch Projectiles	192	FF-13	HBX	Spiked	6 Hrs	550	288
2	12 Jul	3-inch/5-inch Projectile	192	FF-13	RDX	Spiked	6 Hrs	500	260
4	15 Jul	3-inch Projectiles	192	FF-13	TNT	Spiked	6 Hrs	550	288
6	16 Jul	MK 25 Ship Mines	8	Unused-hot melt coating	TNT	Spiked	48 Hrs	700	371
7	22 Jul	3-inch/5-inch Projectile	192	FF-13	RDX	Spiked	6 Hrs	550	288
8	24 Jul	175mm Projectiles	96	FF-13	Comp B	Spiked	6 Hrs	550	288
9	26 Jul	3-inch Projectiles	192	FF-13	HBX	Spiked	6 Hrs	600	316
10	29 Jul	3-inch/5-inch Projectile	192	FF-13	Yellow D	Spiked	12 Hrs	600	316
1-10	Samples taken at the conclusion of test #10 to check for build-up of residue.								
C	1 Aug	None		N/A	N/A	N/A	24 Hrs	700	371
11	9 Aug	MK 25 Ship Mines	8	Unused-hot melt coating	TNT	Spiked	32 Hrs	700	371
12	13 Aug	3-inch/5-inch Projectile	192	FF-13	RDX	Spiked	6 Hrs	550	288
13	15 Aug	175mm Projectiles	96	FF-13	Comp B	Spiked	6 Hrs	550	288
14	17 Aug	3-inch Projectiles	192	FF-13	HBX	Spiked	6 Hrs	600	316
15	20 Aug	3-inch/5-inch Projectile	192	FF-13	Yellow D	Spiked	6 Hrs	600	316
16	21 Aug	MK 54 Depth Bombs	24	Sawed End (Demil)	HBX	Residue	32 Hrs	700	371
17	25 Aug	3-inch/5-inch Projectile	192	FF-13	RDX	Spiked	6 Hrs	550	288
18	27 Aug	175mm Projectiles	96	FF-13	Comp B	Spiked	6 Hrs	550	288
19	29 Aug	MK 54 Depth Bombs	24	Sawed End (Demil)	HBX	Residue	24 Hrs	700	371
20	3 Sep	3-inch/5-inch Projectile	192	FF-13	Yellow D	Spiked	8 Hrs	600	316
11-20	Samples taken at the conclusion of test #20 to check for build-up of residue.								
D	8 Sept	175mm Projectiles	480	FF-13	N/A	N/A	6 Hrs	550	288
21	12 Sept	MK 54 Depth Bombs	24	Sawed End (Demil)	HBX	Residue	24 Hrs	700	371
22	16 Sept	106mm Projectiles	192	Demil Facility	Comp A-3	Residue	5.3 Hrs	550	288
23	18 Sept	175mm Projectiles	96	Demil Facility	Comp B	Residue	6 Hrs	550	288
24	20 Sept	3-inch/5-inch Projectile	192	FF-13	Yellow D	Spiked	6 Hrs	600	316
25	21 Sept	MK 54 Depth Bombs	24	Sawed End (Demil)	HBX	Residue	24 Hrs	700	371
26	25 Sept	MK 25 Ship Mines	5	Unused-hot melt coating- Mines from previous testing	TNT	Old Spike	32 Hrs	700	371
28	29 Sept	106mm Projectiles	192	Demil Facility	Comp A-3	Residue	6 Hrs	550	288
27	1 Oct	175mm Projectiles	96	Demil Facility	Comp B	Residue	6 Hrs	550	288
29	2 Oct	3-inch/5-inch Projectile	192	FF-13	Yellow D	Spiked	6 Hrs	600	316
1-29	Samples taken at the conclusion of test #29 to check for build-up of residue.								
"96"	13 Oct	175mm Projectiles	96	Demil Facility	Comp B	Residue	2 Hrs	550	288
30	15 Oct	175mm Projectiles	480	Demil Facility	Comp B	Residue	6 Hrs	550	288
31	19 Oct	175mm Projectiles	480	Demil Facility	Comp B	Residue	6 Hrs	550	288
32	23 Oct	175mm Projectiles	480	Demil Facility	Comp B	Residue	6 Hrs	550	288
33	26 Oct	175mm Projectiles	480	Demil Facility	Comp B	Residue	6 Hrs	550	288
34	29 Oct	175mm Projectiles	480	Demil Facility	Comp B	Residue	6 Hrs	550	288
1-34	Samples taken at the conclusion of test #34 to check for build-up of residue.								

wax stabilizer. Spiking tests were conducted with 3-inch/5-inch projectiles. The most similar item scheduled for demilitarization soon was a quantity of 106mm projectiles loaded with Comp A-3. The supply of 106mm projectiles with an actual residue of Comp A-3 was ample for verification testing of decontamination of this explosive.

There were no items normally loaded with HBX available for spiked tests. Therefore, 3-inch projectiles were employed for convenience in handling. There was a supply of MK 54 Depth Bomb sawed ends containing residues of HBX suitable for decontamination. Those sawed ends were heavily coated with tar-like asphaltic hot melt. Laboratory tests indicated the explosives were soluble in the hot melt and the hot melt would retain the explosive throughout a treatment cycle that would decontaminate the explosive from a metal surface. After communications with CRREL it was decided that test items coated with hot melt would fail the decontamination process if any hot melt remained in the item upon visual inspection. Therefore, the decontamination criteria for the depth bombs was the essentially complete volatilization of the hot melt.

Although TNT was a common explosive, actual munitions items originally loaded with it were not currently available. Therefore, 3-inch projectiles were used for some early tests to confirm prior data. Later tests for TNT decontamination were made with MK 25 Ship Mines, using new, never loaded casings coated with hot melt. Some were spiked from solution in accordance with present procedures. Others were spiked for earlier work by melting a substantially larger quantity of TNT in the mine casing and rotating it for even distribution. The decontamination criteria for the ship mines was complete volatilization of the hot melt.

No items originally loaded with Yellow D were available for these tests at the HWAD facility. However, due to the large quantity of such munitions

on hand for future disposal, tests were made with 3-inch/5-inch projectiles spiked with Yellow D.

2.3.1.2 Program Participants

United States Army Environmental Center (USAEC)-

USAEC had overall program management for the effort to demonstrate the HGD process, including project direction, coordination, and funding. USAEC also provided technical support on explosives chemistry, and specimens for methods development.

Tennessee Valley Authority (TVA)-

TVA was selected by USAEC to manage and conduct the HGD demonstration project. TVA developed the test plan and carried out the portions of the field demonstration including field sampling, laboratory analysis, evaluation, and reporting. TVA carried out related project management activities of planning, staffing, budgeting, and scheduling.

United States Army Defense Ammunition Center and School (USADACS)-

USADACS supported preparatory work by providing laboratory explosives safety training and information, both for the field activities planned and for guidance in setting up the Test Plan. USADACS led the review of test results and contributed to the establishment of HGD as a potential means of decontaminating explosive residues.

Cold Regions Research and Engineering Laboratory (CRREL)-

CRREL provided information as to explosives and munitions chemistry and characteristics to guide TVA's work on Methods Development in the areas of sampling and analysis of explosive residues.

United States Army Center for Health Promotion and Preventive Medicine (USACHPPM) -

USACHPPM was tasked to conduct environmental sampling activities at the HGD facility. Data will be used to extend the HWAD atmospheric emissions permit to include regular operation of the HGD facility.

Hawthorne Army Depot (HWAD)-

HWAD made the HGD system and related facilities at WADF available for testing and demonstration. HWAD inventories of munitions on the demilitarization account provided representative test items for decontamination. They coordinated between other organizations and the operating contractor.

Day & Zimmerman/Basin Corporation (DZB)-

DZB was the operating contractor for HWAD and WADF. DZB personnel conducted all HGD operations except for the actual sampling and analysis activities. They spiked and positioned test items, operated and maintained the HGD facility and equipment, and handled test items to facilitate access and sampling by TVA.

2.3.1.3 Economics

Operating costs were estimated based on data obtained from records at HWAD and from data gathered during the demonstration period from June through October 1994. These figures apply only to the hot gas decontamination facility at HWAD and are not intended to represent operating conditions or costs at other facilities. The final operating cost figures are expressed in dollars per ton (\$/ton) of metal decontaminated and were calculated using information and data collected during the five operational tests (Tests 30 through Test 34). These tests were full chamber loads of 175mm projectiles, 480 projectiles per test, contaminated with Comp B, and representative quantities expected during production runs. A production run of 480 projectiles, each weighing 115

pounds (52.2 kg), is equivalent to approximately 55,200 pounds (25,038 kg) or 27.6 short tons (25.06 metric tons). Figures D-49 through D-53a indicate arrangement of pallets of projectiles inside the decontamination chamber. Details of the cost estimate are given in Section 5.

2.3.2 Phase II

A conceptual design for construction of a new HGD facility has been prepared by TVA. (Reference #5, TVA Report). That report includes the process design, equipment and facility design, and capital and operating cost estimates.

In the conceptual design process, ambient air is heated in a heat recovery heat exchanger (using thermal oxidizer exhaust gas) and introduced into the decontamination chamber where explosive residues are volatilized into the air stream. The explosives in the air stream are destroyed in a thermal oxidizer by raising the air stream temperature to over 1800°F (982°C) and maintaining a residence time of at least two seconds. Based on this design, it is estimated that metal items can be decontaminated at a cost of about \$50 per short ton (\$55/metric ton).

Also, the retrofit of an existing contaminated waste processor (CWP) to a hot gas decontamination facility has been included. Savings in capital expenditures can be recognized by utilizing the existing car bottom chamber, material handling equipment, and other facilities of the CWP. The retrofit will allow the facility to be used for either process (CWP or HGD).

3.0 ANALYTICAL METHODOLOGIES AND PRELIMINARY ANALYTICAL ASSESSMENT

3.1 Introduction to Analytical Methodologies

Sampling and analysis of the explosive residues left in projectiles after the HGD process and sampling of parts of the HGD unit itself were critical for determining how effective and safe the facility is. In order to ensure high quality analytical results, several activities were performed before the demonstration process began. These activities included determining sampling protocol procedures and ensuring that the analysis methods employed would work for the matrices involved. After sampling protocols were developed, tests were conducted in the field to determine the amount of explosives left in melted out items. Detailed procedures for all analytical activities can be found in Appendix B.

3.2 Development of Sampling Protocols

Several types of items were sampled during the HGD demonstration program, each with a different shape and size. To ensure that each item was properly sampled, several experiments were conducted to determine the best sampling protocols for each. There were also several types of explosives considered during the demonstration program including: TNT, RDX, Comp A-3, Comp B, HBX, and Ammonium Picrate (Explosive D/Yellow D). Tests were conducted to determine the best way to sample these explosives as described below.

3.2.1 Extraction Procedures

Since the HPLC analysis methods employed in the demonstration program utilized water and acetonitrile as the solvent for the mobile phase, it was decided that they would be the ideal solvents to use in the extraction process. Using one of these solvents would eliminate an extraction step in the analysis.

Acetonitrile was considered the solvent of choice since it was more likely to dissolve the nitroaromatics. The solubility of each explosive in acetonitrile was determined by placing into separate 100 mL volumetric flasks approximately 10 mg of TNT, Comp A-3, Comp B, and HBX, all in the form of reclaimed flake. Approximately 50 mL of acetonitrile was added to each flask, which was then swirled until dissolution began to occur. The flask was then made to volume with acetonitrile.

The TNT reclaimed flake dissolved totally in 1-2 minutes. The Comp B reclaimed flake dissolved almost totally in 1-2 minutes, with two clear, waxy plates being left. This was thought to be the wax binder portion of the explosive. The HBX reclaimed flake dissolved more slowly than the TNT and Comp B, and required 4-5 minutes of swirling for near total dissolution. Some dark, waxy plates and some very small grayish sediment were left undissolved. This was thought to be the wax binder and powdered aluminum portions of the HBX. The Comp A-3 reclaimed flake did not dissolve at all, even after an hour of vigorous mechanical stirring. Nor, did the Comp A-3 dissolve in acetone, water, methanol, or methylene chloride. It was decided that this was because Comp A-3 is 9 percent wax binder. However, hexane, to a greater extent, and iso-octane, to a lesser extent, each broke up the flake structure of the compound leaving behind a white powdery sediment (RDX crystals). If acetonitrile, whose density is greater than that of hexane, was then added to the hexane mixture, it sank to the bottom and upon agitation dissolved the RDX crystals.

It was determined for the purposes of the demonstration program that the reclaimed flake forms of TNT, Comp B, and HBX would all dissolve in acetonitrile, but a 50:50 mixture of hexane/acetonitrile would be used to get the RDX in Comp A-3 into solution. For Ammonium Picrate, water was used as the solvent of choice since Ammonium Picrate is an organic salt, with a relatively high solubility in water.

3.2.2 Extraction Efficiencies

Once the solvents were determined, an experiment was conducted to determine the best method of extraction and the efficiencies of each rinse used in the system.

This experiment was performed by weighing two aliquots of Comp B (reclaimed flake form) into separate weighing boats. Each aliquot of Comp B was then transferred from its weighing boat to a labeled 200 mL, precleaned, amber bottle wrapped with aluminum foil (to prevent light exposure). Acetonitrile was used to rinse the surface of the weighing boats into the correct bottle. Using a graduated cylinder, 50 mL of acetonitrile was added to each bottle. The bottles were then sealed with Teflon-lined closures and shaken for several minutes. The contents of the bottles constituted the Comp B spiking solutions for this experiment.

The spiking solutions were to be applied to the interior surface of two iron projectile bodies (3-inch projectiles). These projectile bodies had an outside diameter of approximately 3 inches (8 cm), an inside diameter of approximately 2.5 inches (6.3 cm), a length of approximately 12 inches (3 cm), and threads cut on their upper interior wall for a distance of approximately 1 inch (3 cm). The interior of the projectiles was thoroughly rinsed with acetonitrile and allowed to air dry inside a hood. The projectiles were labeled as #1 and #2 on their exterior surface using a permanent marker. Inside the hood, the shells were placed on their sides with their open ends being elevated approximately 0.5 inches (1.3 cm). A mark, running with the long axis of the projectile, was placed on the top exterior side of each projectile so that degrees of rotation could be estimated. The spiking solutions were pipeted into the projectiles (bottle #1 into projectile #1 and bottle #2 into projectile #2) approximately 15 mL at a time being careful to keep the solution off of the threaded portion of the interior as much as possible. The 15 mL aliquots of spiking solution were allowed to dry and the projectile body was rotated 90 degrees

clockwise each time before more solution was added. This continued until all of the spiking solution had been transferred to the correct projectile body. The bottles used to contain and the pipettes used to transfer the spiking solutions were each rinsed three times with acetonitrile with the rinses going into the correct projectile body as before. The evaporation of the acetonitrile went very slowly. Compressed air, at a low flow rate, was blown into the interior of the projectile through a small diameter Teflon tube to help speed the drying process. The spiking process took 6-7 hours and was done in a dark hood, in a room with minimal light. After all acetonitrile had been evaporated, the projectiles were sealed with labeled #11 green, rubber stoppers (that had been previously rinsed with acetonitrile) and left in the hood overnight. The Comp B was extracted from the projectiles bodies the day after they were spiked. After removing the rubber stoppers, each projectile was extracted three times using steps 1-4 below:

1. The threaded portion of the projectile cavity was rinsed with acetonitrile and the rinse was drained into the projectile cavity. 25 mL of acetonitrile was then added to each projectile and a rubber stopper was hand driven as far into the projectile opening as possible.
2. The projectile was rocked from the vertical to the horizontal position several times, while at the same time rotating the body around its long axis to achieve thorough rinsing of the spiked cavity.
3. After the projectile was set upright, the stopper was carefully removed and rinsed with acetonitrile. The rinse was allowed to fall back into the projectile cavity.
4. Using a large pipette, the acetonitrile in the projectile was transferred to a labeled, foil wrapped, 100 mL volumetric flask. The interior of the pipette was rinsed into the flask.

The spike extractions from each of the two projectiles were then made to volume with acetonitrile, sealed, and thoroughly mixed.

A control aliquot of Comp B reclaimed flake was weighed out in the same manner as that of the spiking aliquots. The control weight was 0.0573 grams. This aliquot was placed in an aluminum foil wrapped 100 mL volumetric flask, made to volume with acetonitrile, sealed and thoroughly mixed.

The two projectile extracts and the control aliquot were each diluted 1 to 1250, prepared for HPLC analysis and run in triplicate. Table 3-1 shows the recovery of each of the samples. Each sample was run in triplicate for comparison.

TABLE 3-1
Recovery Efficiency of Comp B Explosive

Sample Name	µg RDX Known	µg RDX Found	Percent Recovery	µg TNT Known	µg TNT Found	Percent Recovery
Extract 1A	33180	29550	89.1	22120	20663	93.4
Extract 1B	33180	29438	88.7	22120	21275	96.2
Extract 1C	33180	28250	85.1	22120	20950	94.7
Extract 2A	31860	28775	90.3	21240	20963	98.7
Extract 2B	31860	29813	90.3	21240	20763	97.8
Extract 2C	31860	28225	88.6	21240	20688	97.4
Control A	34380	31400	91.3	22920	23675	103.3
Control B	34380	30525	88.8	22920	23050	100.6
Control C	34380	31400	91.3	22920	23763	103.7

To determine the number of washes needed to ensure that all of the explosives were extracted from a given projectile, a projectile was extracted several times and each of the extracts were analyzed separately. This experiment was conducted as follows.

Two aliquots of Comp B were weighed out and individually dissolved in approximately 40 mL of acetonitrile. Aliquot #1 weighed 0.0571 grams, and aliquot #2 weighed 0.0564 grams. A pair of 3-inch projectiles were spiked with the Comp B solutions in the following manner. Each projectile was placed on its side, and a 15 mL aliquot of the spike solution was pipeted into its interior cavity. Solution #1 went into projectile #1 and solution #2 went into projectile #2. The projectiles were lying at an angle such that 15 mL of spike solution reached from the base of the cavity to the bottom of the threaded portion of the interior. The solvent portion of the spike aliquots was slowly evaporated by placing a small airline at the mouth of each projectile and allowing UPC grade air, at 20 psi, to blow into its interior. As the interior of each projectile reached dryness, the projectiles were rotated about their long axis and spiked with more Comp B solution. This continued until all of the spiking solutions were expended and the spike containers and pipettes had been rinsed into their respective projectiles. Next, each projectile was extracted ten times in the following manner:

- 100 mL of acetonitrile was placed into each projectile.
- A stopper was placed firmly into the top of each projectile.
- Each projectile was held with its long axis at a 45-degree angle from vertical and rotated about its long axis while gently agitating the solution; this was done for 2 minutes.
- The stopper was removed and the aliquot of extract solution was placed in a vial wrapped with aluminum foil.

Before analysis, the first rinse of each projectile was diluted by a factor of 1250 and the second rinse for each was diluted by a factor of 50. Table 3-2 shows the analytical results of this experiment in tabular form.

TABLE 3-2
Efficiency of Multiple Washes for Comp B in a 3-inch Projectile.

Rinse No.	Original μg RDX & TNT	μg RDX Found	μg TNT Found	Total μg Found	Percent Recovery of Original
Projectile #1					
1	57100	30981	20911	51892	90.9
2	57100	602	430	1032	1.8
3	57100	26	33	59	0.10
4	57100	5.4	5.7	11.1	0.02
5	57100	5.3	8.0	13.3	0.02
6	57100	1.6	1.5	3.1	0.005
7	57100	1.1	1.3	1.3	0.004
8	57100	1.3	0.5	0.5	0.003
9	57100	0.9	1.4	1.4	0.004
10	57100	<MDL	0.8	0.8	NA
Projectile #2					
1	56400	31510	18697	50207	89.0
2	56400	527	381	908	1.6
3	56400	32	41	73	0.13
4	56400	11	17	28	0.05
5	56400	8	13	21	0.04
6	56400	6	9	15	0.03
7	56400	4	6	10	0.02
8	56400	4	6	10	0.02
9	56400	3	4	7	0.01
10	56400	3	4	7	0.01

Note: Since Comp B contained primarily TNT and RDX it was assumed that the extraction efficiencies for pure TNT and RDX would be similar.

As the results indicate, approximately 90 percent of the explosives are extracted on the first rinse. After discussions with USAEC (Environmental Technology Division), it was determined that one rinse would be sufficient for this project. This decision was made for two reasons. First, since only low quantities of explosives were expected to remain after decontamination, it was decided that any rinses beyond the first would

contain such a small amount of explosive that it would not be detectable. Second, since this was a field operation, there was likely to be some error in recovery, and that error would be of the same magnitude of the material which might be recovered in successive washes.

A two phase solvent system was required to dissolve Comp A-3. It was not feasible to do any extraction efficiency tests since there was not a suitable method for spiking the projectiles with Comp A-3. However, the solvent system used dissolved the Comp A-3 readily and allowed good analytical recoveries of RDX as shown in Table 3-3. In the actual chamber tests, only melted out projectiles were used. Since Comp A-3 contains RDX, it was assumed that the recoveries for Comp A-3 would be comparable to those for Comp B. Thus for projectiles that contained Comp A-3, they were rinsed out using the same procedure as for other explosives using the mixed solvent system.

Table 3-3
Recoveries of RDX From Hexane Acetonitrile Extraction Mixture.

Sample Number	Wt. in grams Comp A-3	Possible µg of RDX ⁽¹⁾	Average µg RDX Found	Average % Recovery
1	0.0111	10101	9029	89.4
2	0.0120	10920	9782	89.6
3	0.0129	11739	0605	90.3
4	0.0100	9100	8207	90.3
5	0.0110	10010	9430	94.2
6	0.0136	12376	11287	91.2
7	0.0133	12103	11640	96.2
8	0.0119	10829	10064	92.9
9	0.0116	10556	9665	91.6
10	0.0123	11193	10417	93.1

Average recovery for all analysis = 91.9%
Standard deviation of all recoveries = 2.20

⁽¹⁾Based on Comp A-3 being 91% RDX

Water was the solvent of choice for the Ammonium Picrate since it performed well in this system. A rinsing experiment was performed, similar to that already described, using Ammonium Picrate as the explosive and water as the solvent.

Two 3-inch projectiles were spiked and rinsed to determine an adequate number of rinses for the removal of Ammonium Picrate. A solution of Ammonium Picrate in acetone (~70mg/75mL) was added to each projectile in 15-mL aliquots. Each aliquot was allowed to dry in the projectile before the next was added. Each projectile was then rinsed sequentially with 100 mL of OmniSolv water five (5) times and submitted for HPLC analysis. Table 3-4 contains the data for the recovery experiment.

Table 3-4
Recovery of 3-Inch Projectiles Spiked With Ammonium Picrate

Projectile #1		
Rinse No.	Ammonium Picrate recovered (mg)	Percent Recovered
1	58.4	90.3
2	0.706	1.1
3	0.0303	0.05
4	0.00623	0.01
5	0.00265	0.004
Projectile #2		
1	61.34	90.3
2	1.083	1.1
3	0.06936	0.05
4	0.04906	0.01
5	0.01001	0.004

Note: Projectile #1 had 64.7mg initially and projectile #2 had 68.0mg initially of Ammonium Picrate.

As the data in Table 3-4 indicate, about 90 percent of the spiked material is extracted after the first wash. About 1 percent is obtained after the second wash and all successive washes are below 0.05 percent.

3.2.3 Wipe Procedures

For the mine bodies tested, it was not practical to rinse them with solvent because of their size and shape. TVA developed a wipe procedure to check for explosive residues on these items. The details of that procedure are given in Appendix B. To ensure that the procedure gave acceptable recoveries, it was tested in the laboratory in the following manner.

A spiking solution was prepared by weighing out 0.0118 grams of Comp B and diluting the dissolved compound to 10 mL in a volumetric flask. The surface to be spiked was a steel disk, approximately 4.5 inches (11.4 cm) in diameter, containing a scribed rectangular area approximately 5.8 cm by 6.6 cm in size. The steel disk was placed in a darkened hood in a darkened room for the spiking procedure. The spiking solution was applied as evenly as possible to the scribed portion of the steel disk with a pipette. Approximately 2 mL of solution was applied at a time and then allowed to air dry before more solution was added. When all of the spiking solution had been applied and dried, the volumetric flask and the pasteur pipette were rinsed three times with a few milliliters of acetonitrile, which was again placed on the scribed area of the steel disk and allowed to dry.

The Comp B was extracted from the steel disk using a gauze sponge (smear) dampened with acetonitrile. The smear was 3 inches by 3 inches (8 cm by 8 cm), 12-ply, sterile, cotton gauze sponge made by Johnson & Johnson®. The smear was wetted with 5 mL of acetonitrile, then excess solvent was expressed from the smear. The smear was wiped in a circular motion around the spiked area of the steel disk twice without lifting the smear. The smear was then folded in half, with the contaminated portions

of the smear to the inside of the fold, and one of the uncontaminated sides used to wipe the spiked area. The smear was then folded lengthwise, with the contaminated portions of the smear to the inside of the fold, and one of the uncontaminated sides used to wipe the spiked area a third time. The smear was then placed in a pre-cleaned 250 mL foil wrapped jar and sealed with a Teflon-lined lid. The steel disk was wiped in the manner described above with a second and third gauze smear. Each smear was placed in a separate 250 mL jar. A method blank was prepared by wetting a gauze wipe with 5 mL of acetonitrile, expressing the excess solvent from the smear, and placing the smear in a foil wrapped 250 mL jar.

To each of the four jars was added one Teflon-coated stir bar and 100 mL of acetonitrile. After resealing the jars, they were placed on a magnistirrer running at low to medium speed for one hour. The extraction solution from smear #1 was diluted 1 to 100 with acetonitrile. The 1 to 100 dilution of smear #1 plus the extraction solutions of smear #2, smear #3, and the method blank were all prepared for HPLC analysis. Table 3-5 contains the results of the analysis.

Table 3-5
Recovery of Comp B From Steel Disk

Smear No.	Total Known μg RDX & TNT	μg RDX Found	μg TNT Found	Total μg Found	Percent Recovery
1	11,800	6,293	4,422	10,715	90.8
2	11,800	11.7	11.6	23.3	0.20
3	11,800	0.7	0.7	1.4	0.01

From the data, a single wipe sample done properly will be sufficient to check for contamination on a nonpourous surface. Since the recovery for one wipe was in the 90 percent recovery range and after discussions with USAEC (Environmental Technology Division), it was determined that this

was sufficient for the same reasons that were described in the Section 3.2.2 dealing with rinses.

3.2.4 Sampling Hot Melt or Flintcoat Items

The MK 54 Depth Bomb and MK 25 Ship Mines tested in this plan were coated with hot melt or flintcoat. Currently, there is no satisfactory method for extracting explosives out of the hot melt material for analysis. Due to the tight schedule of the project, there was not time to develop a suitable extraction method for analyzing explosives in the hot melt. After conversations with USAEC (Environmental Technology Division), it was decided that for any items coated with hot melt which were to be tested, would fail the decontamination process if any of the hot melt was left in the item upon visual inspection. If no hot melt was present on a decontaminated item, then the item was sampled as described above in Section 3.2.3 to ensure that no explosives residue remained.

3.3 Analytical Methodologies

3.3.1 Method 8330

SW-846 Method 8330 was used as the basis for the analysis of TNT, RDX, HBX, and their degradation compounds. There were some slight variations made in the procedure in order to obtain better detection limits and to fit it to the sample types encountered in this project. The detailed procedure and the modifications are in Appendix B. For all analyses in this project, only the parent explosives (TNT, RDX, Ammonium Picrate) were quantified. Degradation products were qualitatively identified, however they were not observed. The TVA Analytical Lab (ALSS) did not run the confirmation column on the analysis as Method 8330 describes. If there were a question as to the identity of a given peak, it was assumed to be the compound of interest and treated as such in determining destruction efficiencies. During the demonstration program, the TVA lab also sent some samples which exhibited questionable peaks to CRREL where they

were analyzed on the confirmation column. All of the peaks that TVA had identified as explosive were confirmed by CRREL.

3.3.2 Ammonium Picrate

The method for Ammonium Picrate analysis was based on a procedure used by Roy F. Weston, which appears to be adapted from a procedure by Goerlitz that was used to analyze picric acid. The TVA lab had some difficulties with the procedure used by Weston. It was not sensitive enough, and there were problems with the buffers precipitating out in the HPLC system. TVA developed a new method in Appendix B. The method is based on the HPLC analysis of picrate ion rather than picric acid. That is, all the picric acid and other picrate compounds that may be in the sample are converted to picrate ion and analyzed as Picrate ion in the HPLC. It should be noted that for large numbers of samples, this procedure is not ideal because the buffers used in the system degrade the column rapidly. This results in frequent column changes and re-calibration. All QA associated with this method are also described in Appendix A.

3.4 Sampling of Air Stream

3.4.1 Explosives Monitoring of the Gas Stream

In order to determine the compounds present in the gas stream prior to entering the oxidizer, TVA sampled the air stream utilizing a procedure based on EPA's Modified Method 5 procedure for air sampling. The procedure used is described in Appendix B. The resin tube from the Modified Method 5 procedure was rinsed with several washings of acetonitrile. The rinsates were analyzed by HPLC. An example chromatogram from a sample extracted from the TENEX®-TA resin during a test decontaminating Comp B explosive (Test 13) is shown in Figure 3-1. The figure indicates that TNT and RDX are present in the gas stream. However it also indicates that there are many other compounds in the gas stream as well. Figure 3-2 is a chromatogram of a standard containing 0.4 ppm of HMX, RDX, 1,3,5-TNB, 1,3-DNB, NB, TNT, 2,4,-DNT. The

Run File : C:\STAR\MODULE03\STR2542.RUN
Method File : Run File Method
Sample ID : 9408055-41A TENAX

Injection Date: 1-SEP-94 10:09 AM Recalculation Date: 7-APR-95 11:52 AM

Operator : Number 1 Detector Type: 9050
Workstation: Bus Address : 3
Instrument : Varian Star Sample Rate : 2.50 Hz
Channel : 1 = 254 nm Run Time : 19.993 min

***** Varian Star Workstation ***** Rev. C 08/20/90 *****

Chart Speed = 0.80 cm/min Attenuation = 4 Zero Offset = 1%
Start Time = 0.000 min End Time = 19.993 min Min / Tick = 1.00

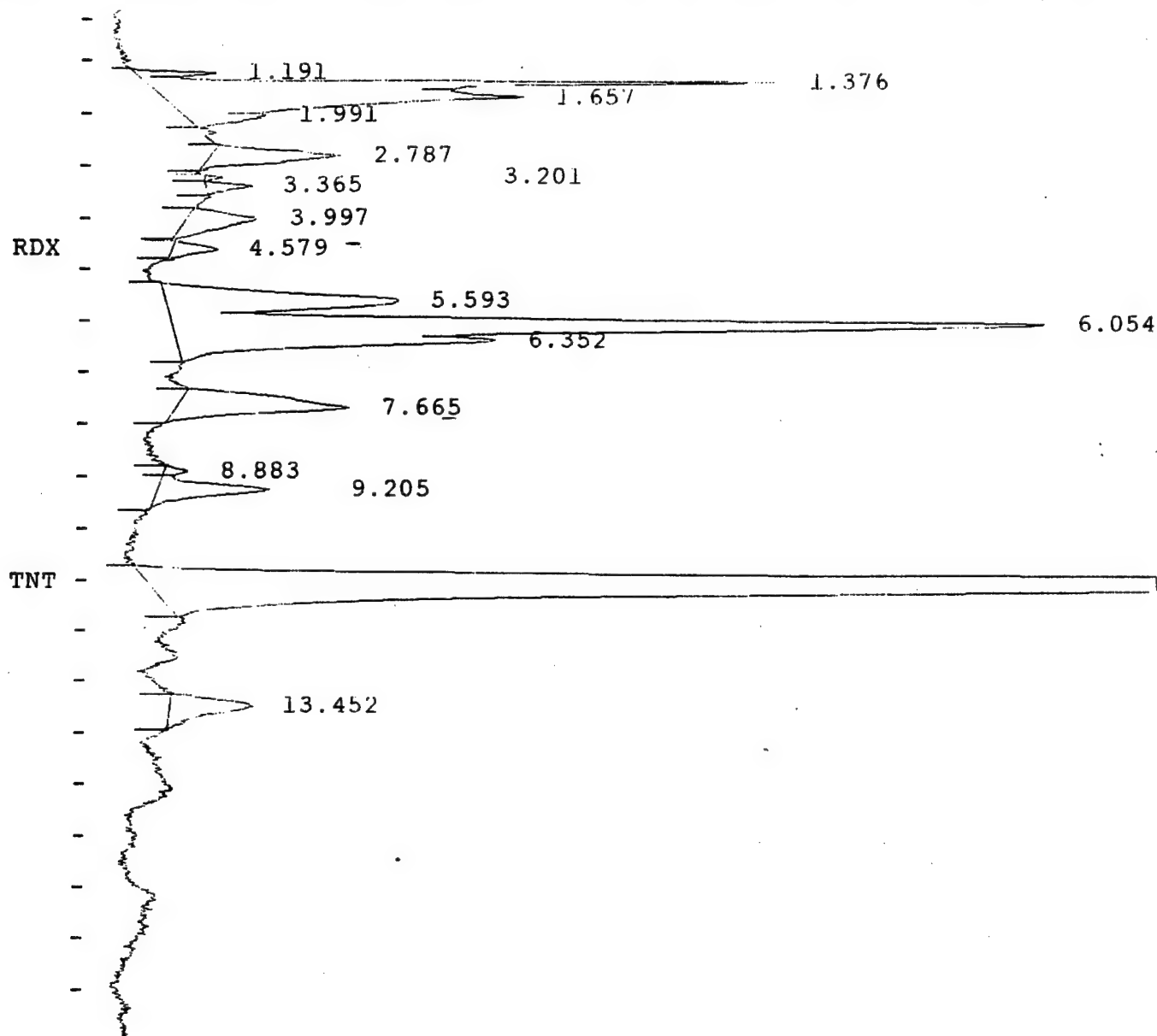


Figure 3-1. Typical Chromatogram of Chamber Exhaust Gases.

Run File : C:\STAR\MODULE03\STR3547.RUN
Method File : Run File Method
Sample ID : 7 EXPL. @ 0.4ppm

Injection Date: 13-OCT-94 4:03 PM Recalculation Date: 7-DEC-94 1:36 PM

Injection : Number 1
Position :
Instrument : Varian Star
Channel : 1 = 254 nm
Detector Type: 9050
Bus Address : 3
Sample Rate : 2.50 Hz
Run Time : 19.987 min

***** Varian Star Workstation ***** Rev. C 08/20/90 *****

Chart Speed = 0.80 cm/min Attenuation = 4 Zero Offset = 15%
Start Time = 0.000 min End Time = 19.987 min Min / Tick = 1.00

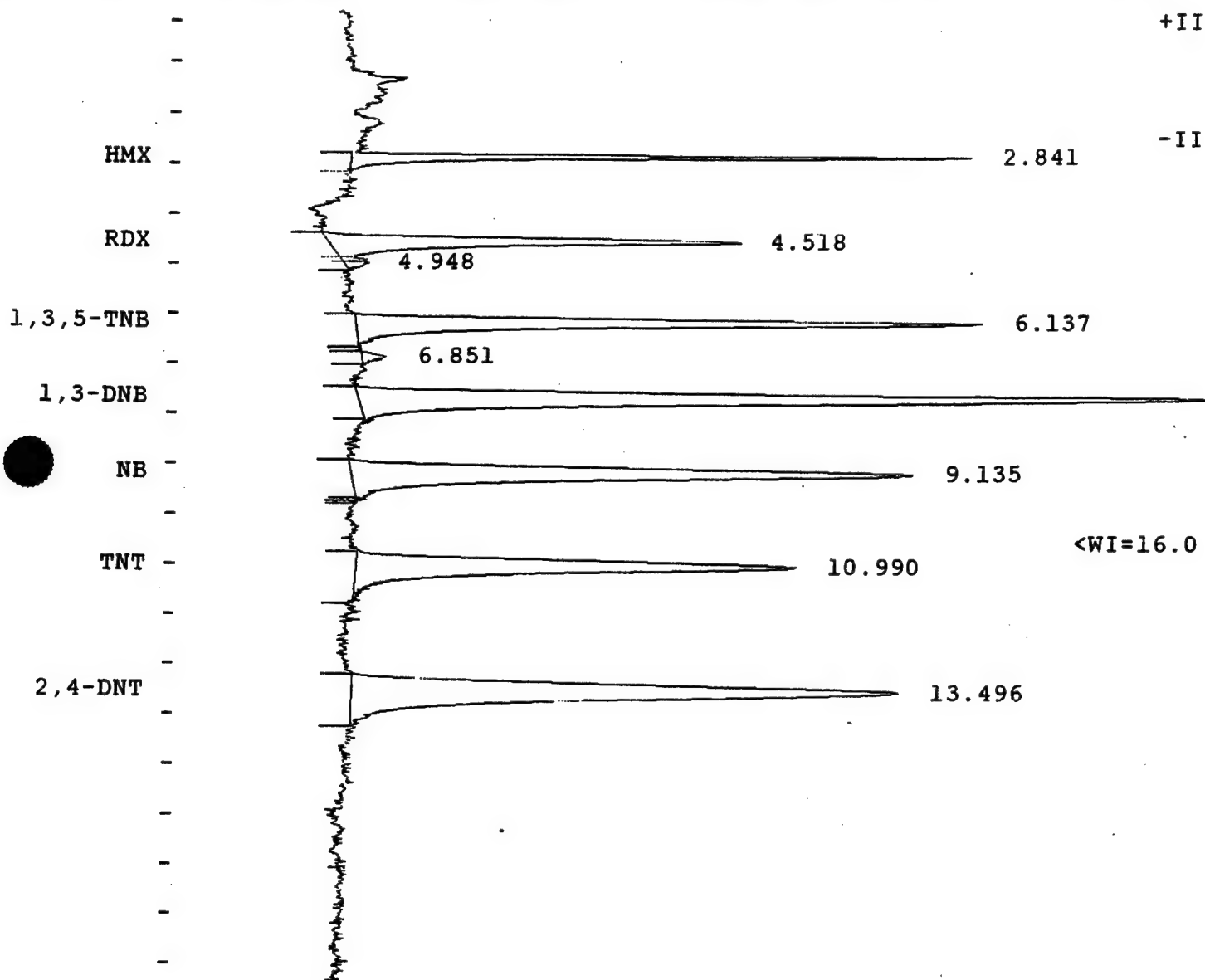


Figure 3-2. Chromatogram of a Standard Containing 0.4 ppm of HMX, RDX, 1,3,5-TNB, 1,3-DNB, NB, TNT, 2,4-DNT in Acetonitrile.

retention time for the breakdown products compare well with some of the unidentified peaks in the TENEX®-TA sample. A comparison of the retention times for the TENEX®-TA peak and the standard are shown in Table 3-6. From the table, it can be seen that there are also many peaks that are not in the standards. These peaks could be due to other thermal decomposition products from explosives (or to other organic compounds that are being driven from the system). These compounds could be from the paint on the projectiles or from lubricants that are used on the railroad car.

Table 3-6
Comparison of TENEX®-TA Extract Retention Times
With 0.4 ppm Multipoint Standard

Compound	Standard Retention Time	TENEX®-TA Retention Times
		1.191
		1.376
		1.657
		1.991
		2.787
		3.201
		3.365
		3.997
RDX	4.518	4.579
		5.593
1,3,5-TNB	6.137	6.054
		6.352
1,3 DNB	7.659	7.665
		8.883
		9.205
TNT	10.990	11.009
2,4 DNT	13.496	13.452

It was not possible to get a good material balance between the amount of explosives spiked into the system and the amount recovered in the TENEX®-TA tube. There are two probable reasons for this discrepancy. First, there was some difficulty with breakthrough in the TENEX®-TA tube due to the relatively high concentrations of explosives in the gas stream. The system was modified in an attempt to remedy the situation. These are

described in Appendix B. However, these modifications did not greatly enhance the procedures. Second, since there appear to be many types of breakdown products present in the gas stream, it was impossible to be assured that all of the explosive material used in the system was accounted for. After discussions with USAEC, it was determined that the information gained from gas sampling was useful but not critical to the projects success. And since it appears that it would have taken a significant amount of time to work out all of the problems, which the program schedule would not allow, it was decided to discontinue gas sampling for this particular demonstration program.

3.4.2 Continuous Emissions Monitor (CEM) Monitoring of Air Steam

TVA rented CEM analyzers from CAE Incorporated, Palatine, Illinois, to monitor the concentrations of CO, CO₂, NO_x, and THC in the gas stream prior to the oxidizer. CAE installed the analyzers on the HGD facility and conducted initial calibration of the systems. The systems were then set up to calibrate on a standard gas automatically every day at 2 a.m. If a problem occurred, CAE was contacted and their service representative would visit the job site and correct the problem. Data points were taken at one-minute intervals for all of the gases of interest from the time the system was started until the system was shut down. The data points were collected in a datalogger (laptop computer) and stored on computer disk. The data was imported into Lotus 123[®] and plotted. All of the plots for the gas analysis are in Appendix F.

3.5 Preliminary Field Experiments

3.5.1 Determination of Residues in Melted Out Items

To determine the amount of residue left in melted out items, TVA conducted an experiment with melted out 175mm projectiles. Three projectiles were subjected to twelve sequential 500 mL rinses of acetonitrile. During two of the rinses, the projectile was brushed with a test tube brush to remove large chunks of remaining material. Each of the

rinses from the projectiles were returned to TVA, ALSS, for analysis. Each rinse was analyzed separately, and then the rinses were summed to get the total amount of explosives present in the projectiles. The results for the three projectiles were 19.4g, 16.5g, 42.5g, from each of the projectiles, respectively. It should be noted that the projectile containing 42.5g had explosive residue in the threads where the nose plug was inserted; the others did not. After visual inspection of several more melted out items, it was determined that very few had large amounts of residue in the threads. As a result, it was decided that a reasonable value to use for the rest of the experiment for the amount of residue remaining in the projectiles was approximately 20g.

For the spiking experiments, 20g was used for the 175mm projectiles. Since 175mm projectiles (melted out) were the only size available (during spiking), it was assumed that the residue concentration would be linear in proportion to the internal surface area of the projectile, thus the amount used for the other sized projectiles was scaled appropriately to their internal surface area.

Table 3-7
Explosive Residue Remaining in Melted Out Projectiles

Projectile	Residue Remaining (g)	Surface Area (sq cm)
175mm	20.0	1,923
5-inch	7.0	632
3-inch	2.2	202

3.5.2 Dealing With Hot Melt or Flintcoat Items

As mentioned earlier, hot melt coated items could not be analyzed. For these items to be considered decontaminated, all of the hot melt must be removed. An experiment was performed to determine the volatility of the hot melt as a function of both time and temperature.

Six small weighing dishes were weighed, then 1 gram of hot melt was weighed into each weighing dish and each dish numbered. The oven temperature was adjusted and allowed time to reach set point, then the six dishes containing the coating were placed in the oven with #6 in the back of oven and #1 at the front. After 8, 16, 24, 32, 40 and 48 hours, one dish was removed, cooled, and weighed. The results of the tests are shown in Table 3-8.

Table 3-8
Percentage of Hot Melt Volatilized at a Given Time and Temperature

Set Point Temp. (°F/°C)	Time (Hours)					
	8	16	24	32	40	48
500/260	4.9	4.7	1.4	3.2	11.5	12.1
550/288	11.5	16.2	20.0	23.2	28.1	29.8
600/316	23.3	29.7	52.7	61.2	67.9	67.9
650/343	33.6	40.9	60.5	70.5	91.1	86.8
700/371	43.9	59.5	82.8	99.4	99.9	99.9
750/399	72.1	99.7	99.97	-	-	-
800/427	99.9					

From these results, it was decided that the tests involving hot melt or flintcoat items would be run at temperatures at or above 700°F for at least 24 hours. As discussed above, It was also decided that only if there were no visible signs of hot melt or flintcoat on the items that had gone through the process, they would be sampled to verify that no explosive residue remained.

4.0 RESULTS

4.1 Overview

This demonstration program examined a number of variables (treatment temperature, treatment time, explosive type, and munition type) to determine the efficacy of HGD in destroying residues of explosives from munitions. These variables constituted the basic description of the decontamination regimen; what was being treated to remove which contaminant and under what conditions. The effectiveness of explosives removal from the test items was based on the absence or presence of explosive residue after treatment, and if present, its quantity.

The actual sequence, test item, explosive compound, source of test items, condition, and operating parameters of each test is shown in Table 2-2.

The temperature of the test items, chamber, and other points in the system was measured every 15 minutes unless otherwise noted. The composition of the chamber and thermal oxidizer stack (Tests 31-34) exhaust gases was monitored. Extraction samples were taken from the projectile test items and surface wipe samples were taken from the ship mine and depth bomb test items. Wipe samples were also taken at various locations inside the chamber, inside the ductwork, and the I.D. fan blades. Heatup and cooldown times for the system were noted.

Initially, all projectile test items had been processed through the WADF flashing furnace (FF-13) located in Building 117-3. These projectiles were spiked with a known amount of explosives and then processed through the HGD facility. After treatment, they were sampled and analyzed to determine and quantify the presence of explosives. Based on these analyses, a DRE was calculated with the pass/fail criteria being that the DRE must be at least 99.99 percent. Other tests utilized melted out projectiles which prevented the determination of the amount of explosive residue

present in each projectile. However, based on the previously performed baseline experiment (see Section 3.5), it was determined that the minimum amount of explosive residue present in melted out 175mm projectiles was approximately 20 grams and in cases where the fuse threads were covered with residue, the explosive content of these projectiles could be as high as 40 grams. By using the conservative value of 20 grams of residue present in each 175mm projectile (2.2 grams in 3-inch and 7.0 grams in 5-inch projectiles based on the proportional internal surface area of each projectile), DRE were calculated for all of the melted out items.

During this demonstration program, 3091 spiked or melted out test items were treated by the HGD process, with 714 test items sampled and analyzed. Of those 714 samples, only 8 did not meet the DRE requirement which resulted in a demonstration program acceptance rate of 98.9 percent. Of those 7 failures, 2 were achieved during Test 2 which was the first RDX test performed at 500°F (260°C). This temperature was determined to be too low and all subsequent RDX/Comp A-3 testing were performed at 550°F (288°C). Also, 5 failures were from Test 5 which was the first HBX test performed at 550°F (288°C). Again, this temperature was determined to be too low and all subsequent HBX testing was performed at 600°F (316°C). Taking this into account, while performing testing at the proper temperatures, only one failure occurred for an acceptance rate of 99.99 percent.

Discussion of the results from chemical analysis of samples from the test items, chamber and ductwork surface wipes, insulation, and exhaust gases are given in this section. Average temperature data and CEM trends are also included in this section. Data tables showing the results from sampling and figures of temperature and CEM profiles from all the tests are presented in Appendix F.

Results from the explosives and items tested are presented in this section generally in the same sequence as they were first tested and are listed in Table 2-2. Details of all tests are included in Appendix F. Each explosive listed is discussed in so far as possible under the following categories:

- General Observations
- Projectile Extraction Samples
or
- Ship Mine/Depth Bomb Wipe Sampling
- Chamber Wipe Samples
- CEM Trends
- Gas Sampling
- Other Results as Appropriate

4.1.1 General Observations

This section contains a listing of the tests involving a particular explosive, particulars concerning arrangement on railcar or placement inside the chamber and other information pertaining to the explosive. Figures, Tables, and Photos are referenced to provide additional details.

4.1.2 Projectile Extraction Samples

Extraction samples were taken from twenty-four projectiles per test after they had been removed from the chamber and allowed to cool. Solvent was added to the projectile, the projectile plugged, and then rolled for 15 minutes to promote rinsing (Photo E-7). The sampling procedure was the same for both spiked and melted-out projectiles. The extraction procedure is described in Appendix C.

Comp A-3 and Comp B explosive residues were decontaminated from 106mm and 175mm projectiles, respectively, to a DRE of 99.999 percent at treatment times of 6 hours at 550°F (288°C).

Yellow D (Ammonium Picrate) explosive was decontaminated from spiked 3-inch and 5-inch projectiles to a DRE of 99.999 percent at treatment times of 6 hours at 660°F (316°C).

4.1.3 Ship Mine/Depth Bomb Wipe Samples

Wipe samples were taken from twenty-four areas of the sawed-off base ends of the MK 54 Depth Bombs, sixteen areas of the sawed-in-half MK 25 Ship Mines, and twelve areas of the whole MK 25 Ship Mines. Gauze wipes saturated with solvent were applied directly to the sample area to make the extraction. Wipe sampling techniques are in Appendix B and described in Section 3.0.

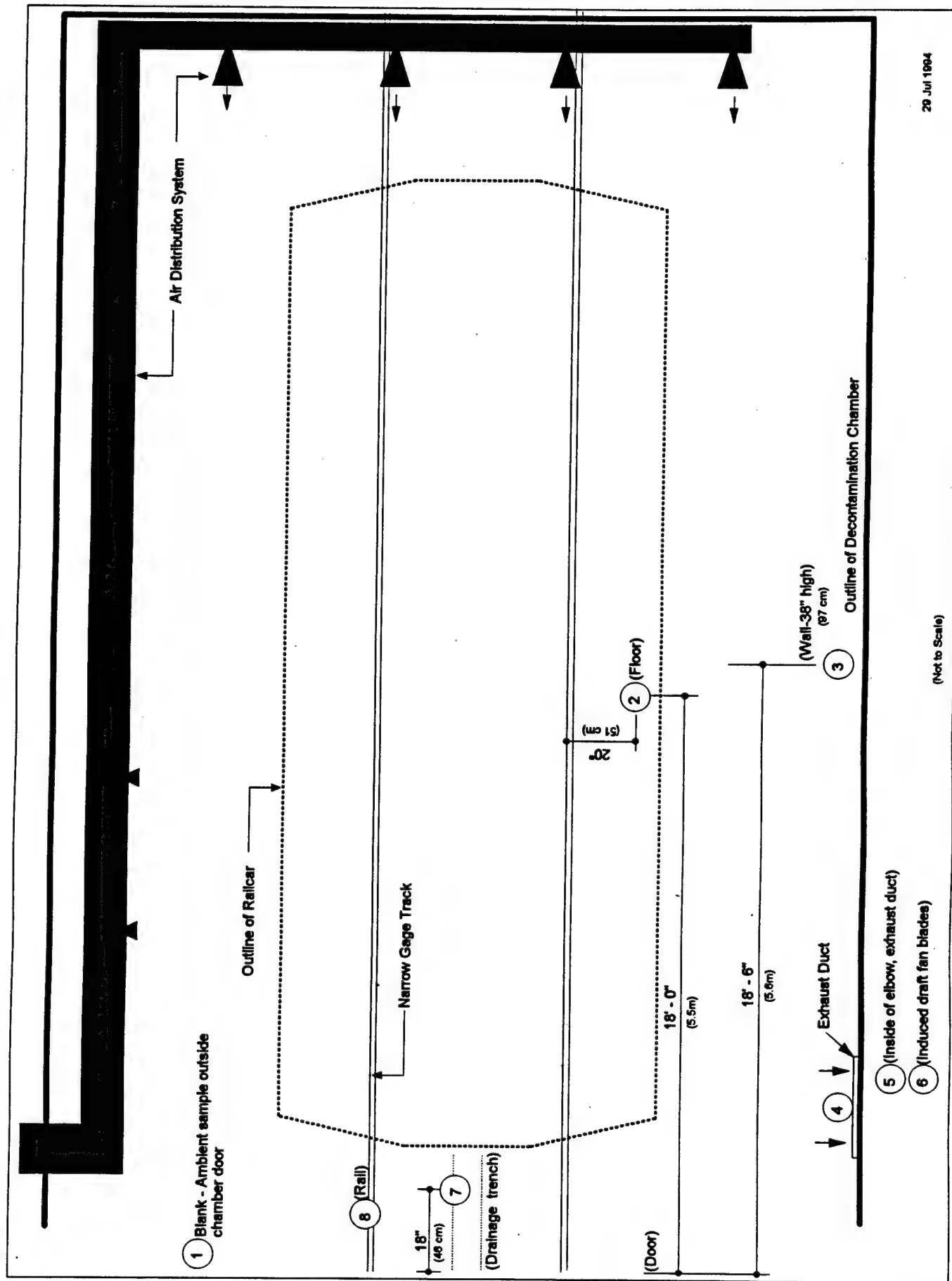
HBX explosive residue was decontaminated from MK 54 Depth Bombs to microgram quantities at treatment times of 24 hours at 700°F (371°C).

TNT explosive was decontaminated from spiked MK 54 Ship Mines to a DRE of 99.999 percent at treatment times of 32 hours at 700°F (371°C).

4.1.4 Chamber Wipe Samples

Wipe samples were taken from several locations inside the chamber, from the chamber exhaust duct, and from one of the induced draft fan blades. The sample locations for the chamber wipes are indicated in Figure 4-1 and were located as follows:

- Floor - 18 feet (5.5 meters) inside the door, 20 inches (51 centimeters) from inner edge of right hand rail.
- Wall - 18.5 feet (5.6 meters) inside the door, 38 inches (97 centimeters) vertically up from the floor on right side of chamber.
- Rail - Left hand rail, just inside door opening.
- Exit Duct - Around inner lip of exit duct.
- Concrete Draining Trench - Left wall of trench 18 inches (46 centimeters) from door, 4 inches (10 centimeters) below floor level.



29 Jul 1994

(Not to Scale)

Figure 4-1 Location of Chamber Wipe Samples

- Exhaust Duct - Inside of elbow, across from sampling nozzle.
- Induced draft fan - Fan blade (marked)

All samples were taken after the chamber had cooled and the test items were removed. The sample area at each location was 25 square inches (161 square centimeters). The wipe procedure is described in Appendix B.

The chamber wipe test results were evaluated by comparing them with the results from the regular test items on a common basis of explosive residue remaining per unit surface area (mg/sq cm). It was reasoned that if a certain level of residue per surface area was acceptable for the test items on the basis that the DRE was greater than 99.999 percent, then a similar level of residue per surface area for the chamber wipes would be acceptable with regard to personnel and operational safety. In general, it can be stated that all chamber wipe test results were acceptably low on the basis of this comparison. All of the chamber wipe test results were less than 1E-04 mg/sq cm. All test items with results less than this figure had DRE values greater than 99.999 percent.

Explosive residues were detected in 42 of the 256 chamber wipe samples (8 samples per test, 32 tests). The quantity of residue in the 42 samples was similar to the quantities detected in the test item samples. Since the results varied randomly from test to test with no identifiable trend, it appears there was no accumulation of explosive residue.

Accumulative chamber wipe samples were taken after tests 10, 20, 29, and 34 to determine if there was an accumulation of explosive residue during operation of the system. The wipe samples were taken at generally the same locations as described above except the accumulative samples were offset from the test wipe locations. This allowed the ability to observe any long term accumulation of the explosives.

In the accumulative chamber wipe samples, explosive residues were detected in eight of thirty-two samples with three of the residues found at the "rail" sample location. The average quantity of residue for the eight samples was $1.1269\text{E-}05$ mg/sq cm which is similar to the surface area amounts detected in the projectile extraction samples. It appears there was no long term accumulation of explosive residue from test to test since there was no identifiable buildup of explosives over time.

It appears that any explosive residue left in the chamber in the vapor phase after decontamination of the test items may deposit on the chamber surfaces randomly during the cooldown of the system. Explosive residues were detected at the "rail" wipe sample location sixteen times; the "wall", the "concrete draining trench", and the "exit duct" sample locations four times each; and the "floor" sample location two times during the thirty-two tests. It is thought that personnel and equipment entering the chamber to remove the railcar and thermocouple probes could have been a source of contamination at the "rail" sample location. Explosive residues were not detected at any sample location (rail) for more than three consecutive tests. In the tests with three consecutive detectable quantities of explosive residue there was no accumulation of explosive material; the quantity of residue increased and decreased. The amount of explosive residue detected was very small ($3.34\text{E-}06$ to $6.38\text{E-}05$ mg/sq cm) as demonstrated throughout the results section. When the system was restarted, the microgram quantity of explosive would revolatilize as the temperature increases.

4.1.5 Continuous Emissions Monitor (CEM) Trends

A CEM was installed at a sample point on the chamber exhaust duct. It provided a continuous, time-keyed record of THC and inorganic combustion products such as NO_x , CO, and CO_2 . The CEM data showed the composition of the chamber atmosphere including the thermal oxidizer flue gas and the oxidation products of the explosive compounds.

CEM test data showed well defined increased NO_x and CO activity for Comp B, HBX, and Ammonium Picrate explosives. This activity was not as well defined for Comp A-3 and TNT explosives. Increased CO activity was observed during volatilization of the hot melt when testing MK 25 Ship Mines and MK 54 Depth Bombs. CEM activity is discussed in Appendix F in detail for each individual test.

4.1.6 Gas Samples

4.1.6.1 Chamber Exhaust Duct

Gas samples were taken from the chamber discharge duct during several tests by TVA personnel (Photo E-8). The samples were taken by a technique based on EPA Modified Method 5. Samples of the absorptive resins and impinger solutions were analyzed for explosives and related compounds.

Laboratory results of gas samples taken from the chamber exhaust duct indicated the presence of TNT degradation products. The presence of degradation products indicate the most likely mechanism of decontamination is thermal degradation not volatilization of the explosives.

Explosive residues were also detected in the "exhaust duct" wipe sample location seven times and in the "induced draft fan" sample location two times during the demonstration test program. The residues were not detected at either sample location for more than two consecutive tests with no accumulation in the microgram quantities of explosive residue. The amount of explosive residue was very small ($2.99\text{E-}06$ to $7.31\text{E-}05$ mg/sq cm)

4.1.6.2 Thermal Oxidizer Exhaust Stack

USACHPPM provided exhaust stack sampling (Photo E-9) for environmental considerations. Their sampling and analysis data will be used to modify the existing facility air permit before the system becomes operational for full

scale production. Emissions of THC, CO, NO_x, SO₂, particulates and metals were measured. The SO₂ emissions were produced by the diesel fuel used to fire the thermal oxidizer.

Results from sampling of the stack during full chamber load tests of melted-out 175mm projectiles containing Comp B explosive showed emission rates of total hydrocarbons, SO₂, NO_x, CO, particulates, and metals significantly below state of Nevada allowable limits.

4.1.7 Insulation Samples

Insulation samples were placed inside the chamber to determine if they would accumulate explosives residues during operation of the system. Results obtained were used in TVA's HGD conceptual design report (Reference #5, TVA Report). Two types of insulation, a 1-inch thick Kaowool blanket and a pre-fired Pyroblock YR, were tested. A sample of each type was placed at the following locations:

- Railcar midpoint - on mid point of railcar in airstream
- Railcar rear - on door end of railcar in airstream
- Wall midpoint duct side - on wall of chamber, waist high, exhaust duct side, midpoint of railcar,
- Ceiling - near ceiling, midpoint of railcar
- Wall near door opposite duct - on wall of chamber opposite exhaust duct, door end, out of airstream
- Duct - outside of exhaust duct

The samples were first installed in the chamber during Test 8 (Photos E-13, E-14, and E-15). Portions (1 inch square by insulation thickness) of the samples were removed for analysis after Tests 8, 12, 18, 23, 27, and 34. Samples taken after these tests were an accumulation of residues from all earlier tests.

Explosive residues were detected in only six of seventy-two samples with no sample location having more than one detectable result. The average quantity of residue for the six samples was 2.8460E-05 mg/cubic cm.

Since the results varied randomly from test to test with no identifiable trend, it appears there was no accumulation of explosives residue. Detailed results are presented in Tables F-79 through F-84 in Appendix F. These results are true regardless of the explosive under consideration and under all test conditions.

4.2 Explosives Decontamination

4.2.1 Test B

General Observations

This test was the first in the demonstration series and was conducted on 96 inert 175mm projectiles in a railcar configuration as shown in Figure 4-2 and Photo 4-12. The test was performed to establish the temperature profile for a typical railcar configuration of items to be tested and to establish the maximum practicable operating temperature of the system. The control temperature was set at 500°F (260°C) for 6 hours then increased to 850°F (454°C) for 24 hours. The temperature profile is shown in Appendix F as Figure F-1.

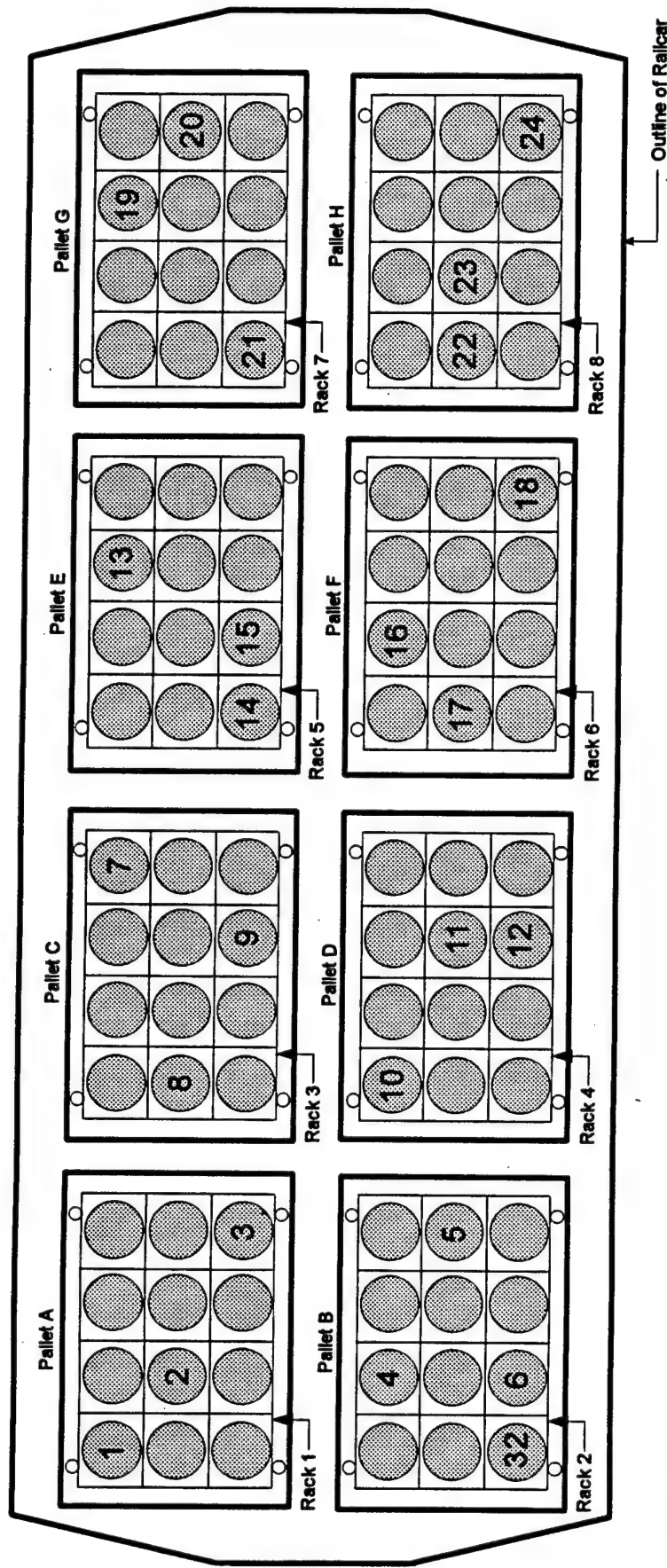
Since the 850°F (454°C) temperature could not be attained, 700°F (371°C) was accepted as the maximum operating temperature and was recorded as the operating temperature.

The temperature data from the first six hours of the test was used to locate the temperature control element to the area in the chamber which was the slowest to reach the desired temperature. The temperature control element remained fixed for all the railcar tests with explosives. Items in other areas of the chamber were likely to be treated at a higher temperature than the nominal test conditions.

Total Weight, 96 Projectiles = 11,040 lbs (5,008 kg)

175mm Projectiles (items from FF-13)

Inert projectile added for thermal mass



See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-6 for rack placement on pallets

Test B
(Not to Scale)

22 June 1994
6 Hours at 500° F / 260° C &
24 Hours at 700° F / 371° C

Figure 4-2 175mm Projectiles (Inert)

The temperature data from the remaining twenty-four hours of the test was used to determine the nominal sustained operating temperature of the chamber, and would be used during tests decontaminating Yellow D and hot-melt coated munitions

Projectile Temperatures

The coldest projectile temperature was located in the projectile nearest the exhaust duct. The control thermocouple for subsequent tests was placed inside an inert 3-inch projectile, suspended approximately railcar high, on the right wall of the chamber as shown in Photo 4-1 and 4-2.

The control temperature did not significantly exceed 700°F (371°C) even after 30 hours of continuous heating. The system had reached but not sustained 800°F (427°C) during proveout tests by DZB earlier. A number of reasons for the shortfall in temperature were considered, including the difference in railcar configuration and thermocouple placement, but none could be proven.

Chamber Wipe Samples

Explosives residues were detected in all of the samples taken at the conclusion of Test B. Since the average quantity of residue was only 1.8543E-05 mg/sq cm, and no explosives had been in the chamber for this test, the error was probably a TVA procedural error which was corrected in subsequent tests. This particular source of contamination was not identified and did not re-occur in later tests.

CEM

The CEM equipment was not operational during this test.

Gas Sampling

Gas samples were not collected during this test.

4.2.2 Explosive: TNT

4.2.2.1 3-Inch Projectiles

General Observations

TNT contaminated projectiles were decontaminated in Tests 1 and 4. Each test was performed with 192 3-inch projectiles in racks placed on a railcar. Each railcar contained 168 inert projectiles for thermal mass and 24 spiked projectiles (as described in Appendix B) which were internally monitored for metal temperature. They were arranged on the railcar generally as shown in Figure 4-3 and in Photo 4.7. The location of the spiked projectiles in the racks was varied in the two tests to provide temperature monitoring and sampling over as many locations on the railcar as possible.

Projectile Extractions

Test 1 was conducted at a treatment time of 6 hours and a control temperature of 500°F (260°C). The chemical analysis of the extracted samples revealed substantial residues of TNT and RDX. Since this was not credible for projectiles which had been spiked with TNT only, this test was declared invalid and the analytical results were unusable.

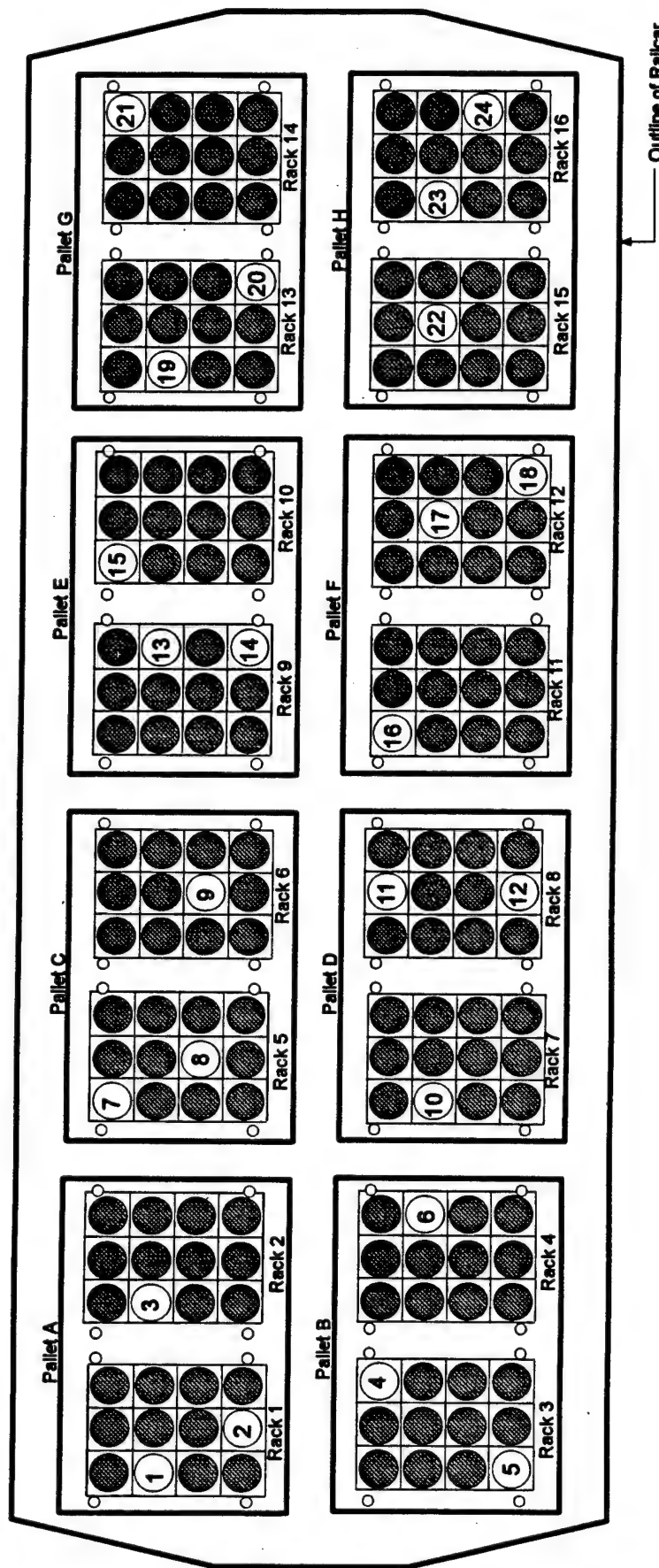
After sampling the nose plugs used to close the projectiles during sample extraction, levels of 0.253 mg of RDX and 0.076 mg of TNT were found. It became necessary to use new plugs (or decontaminated used plugs from the existing flashing furnace; FF-13) for future tests with 3-inch and 5-inch projectiles.

Later, when Test 4 was conducted at a control temperature of 550°F (288°C) for 6 hours, all projectiles were decontaminated to a DRE of 99.999 percent or better. The average temperature profile for the 6 hours of Test 4 ranged from 553 to 595°F (289 to 313°C) and is shown in Figure F-6.

Total Weight, 192 projectiles = 1,728 lbs (784 kg)

3-inch Projectiles (Items from FF-13)

- ④ Spiked 3-inch projectile sampled
- Inert 3-inch projectile added for thermal mass



Door Opening

Outline of Railcar

(Not to Scale)

See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-3 for rack placement on pallets

Figure 4-3 3-inch Projectiles Spiked with TNT

Chamber Wipe Samples

Explosive residues were detected in two of eight samples with an average quantity of 3.0586E-05 mg/sq cm, which was an order of magnitude greater than the projectile extraction samples.

CEM Trends

When compared to baseline levels from Test B2, Test C and Test D (no explosives) (Figures 4-16, 4-17, and 4-18, respectively), there were no appreciable changes in CO, CO₂, THC, and NO_x. However, the NO_x level increases and decreases as the temperature increases and decreases. This is likely due to NO_x in the combustion gases from the fuel of the thermal oxidizer.

Gas Samples

No gas samples were collected during these tests.

4.2.2.2 MK 25 Ship Mines

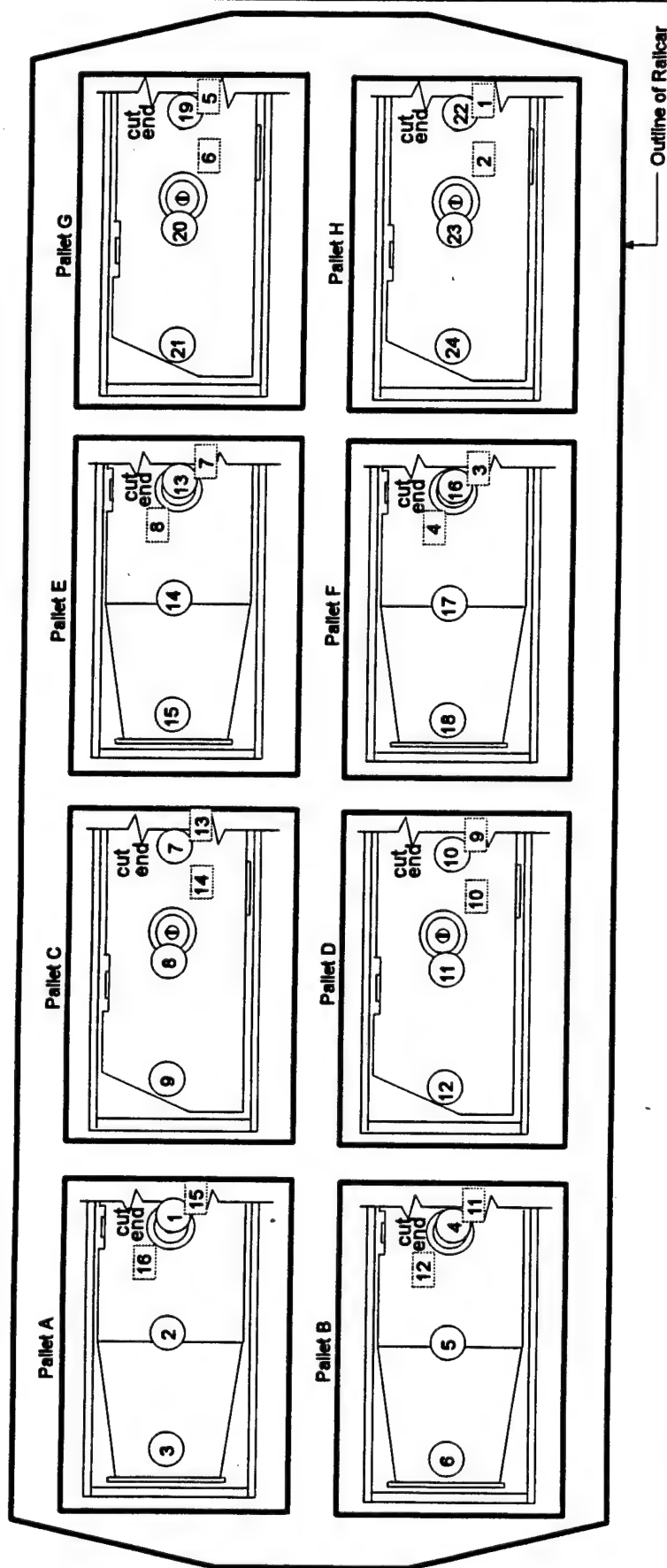
General Observations

TNT explosive spiked over hot melt was decontaminated in Tests 6, 11, and 26. The decontamination criteria for these tests are described in Section 3.2.4. Tests 6 and 11 were conducted with four MK 25 Ship Mines, sawed in half, and placed on pallets on a railcar. They were arranged on the railcar as shown in Figure 4-4 and in Photo 4.8. The temperature probes and sampling locations were the same in both tests. Test 26 was conducted with 2 halves of an inert ship mine for thermal mass (treated in Test 11) and 3 whole ship mines on pallets on a railcar. They were arranged on the railcar as shown in Figure 4-5 and in Photo 4.9. The ship mines in Tests 6 and 11 were spiked (48 grams per mine) by TVA a few days before the tests. Photos E-11 and E-12 show MK 25 Ship Mines internals before and after

Total Weight, 4 Mines = 2,860 lbs (1,297 kg)

MK 25 Ship Mines (Unused mines - internals coated with hot-melt)

- 5 Thermocouple Number and Approximate Location
- 1 Sample Number and Approximate Location



(Not to Scale)

See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-8 for mine placement on pallets

Figure 4-4 MK 25 Ship Mines Hot-Melt Coated Internals and Spiked with TNT

HGD Test B2

Empty Chamber w/Railcar

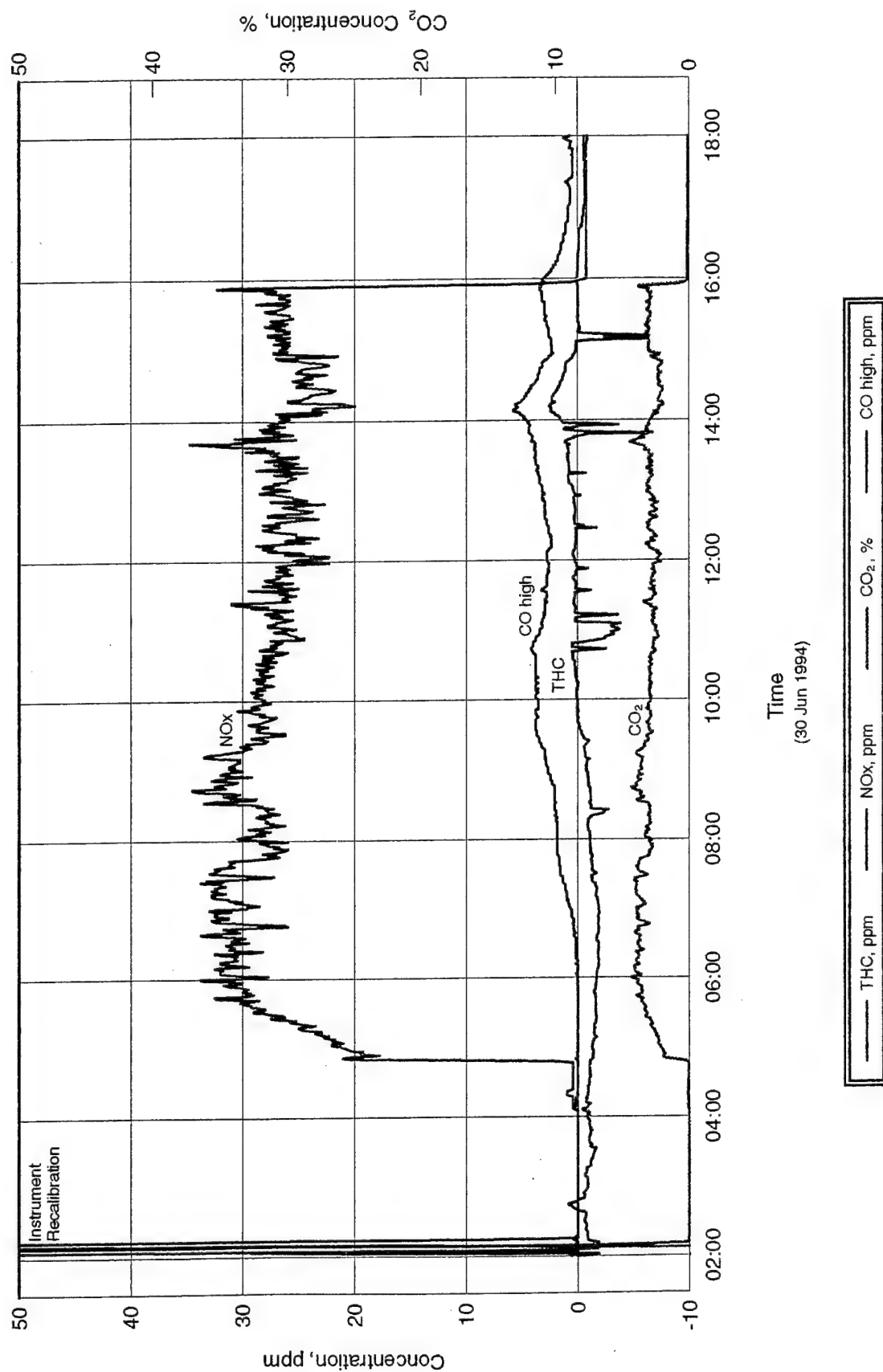


Figure 4-16 Test B2 - Baseline CEM Profile - 1 minute Intervals

HGD Test C

Empty Chamber Decontamination

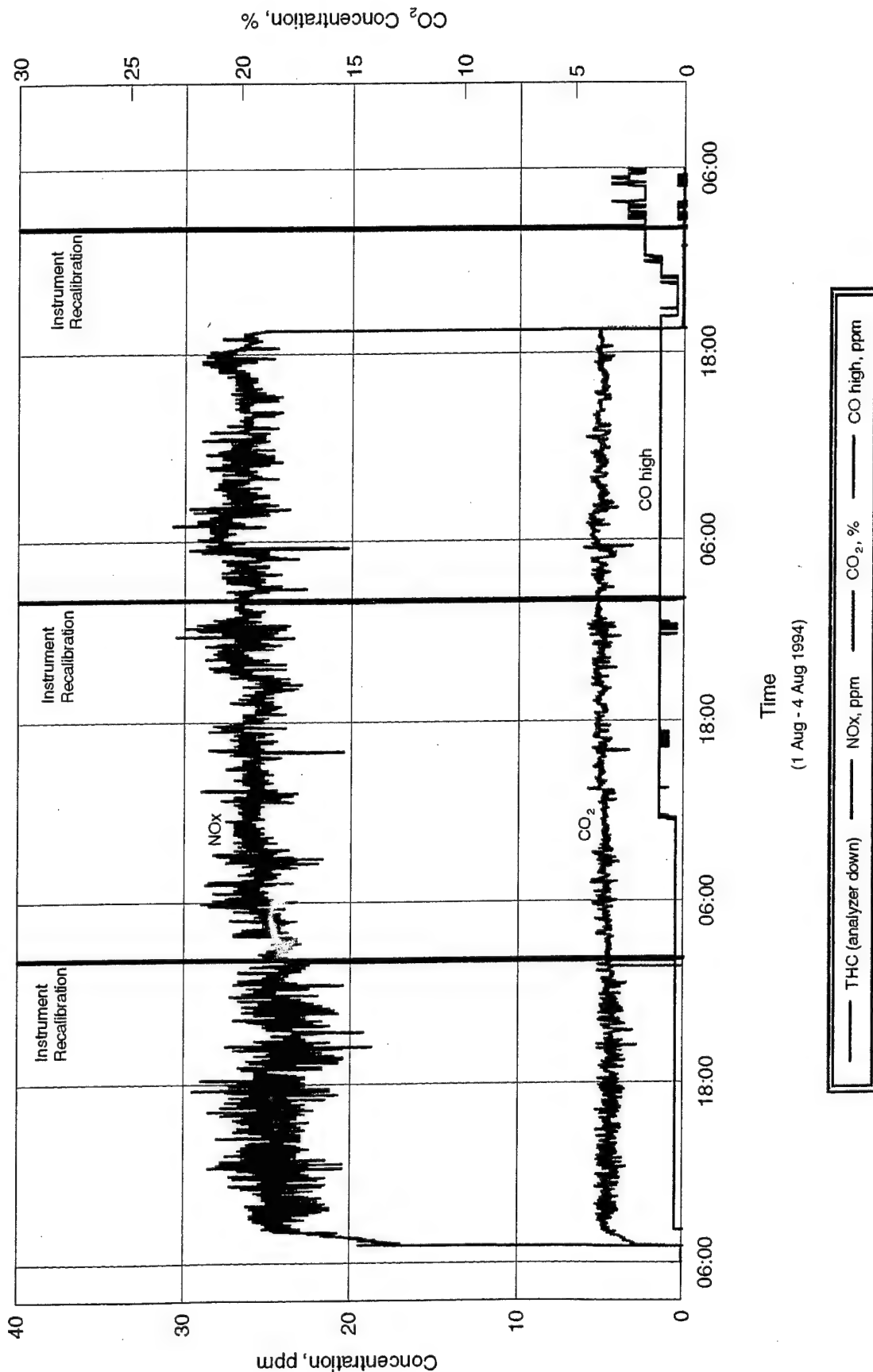


Figure 4-17 Test C - Baseline CEM Profile - 1 minute Intervals

HGD Test D

175mm Projectiles - (480 Inert Rounds)

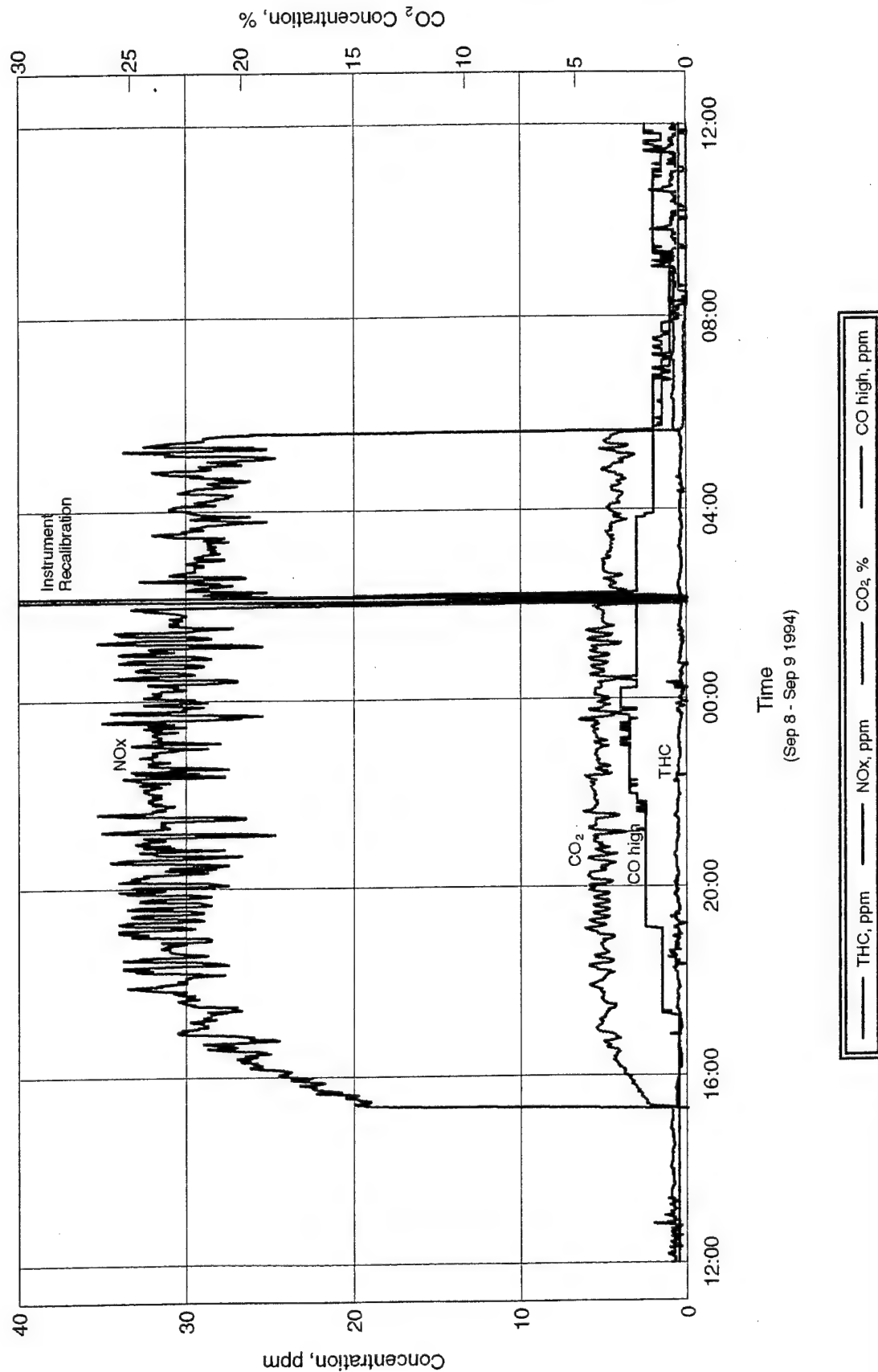


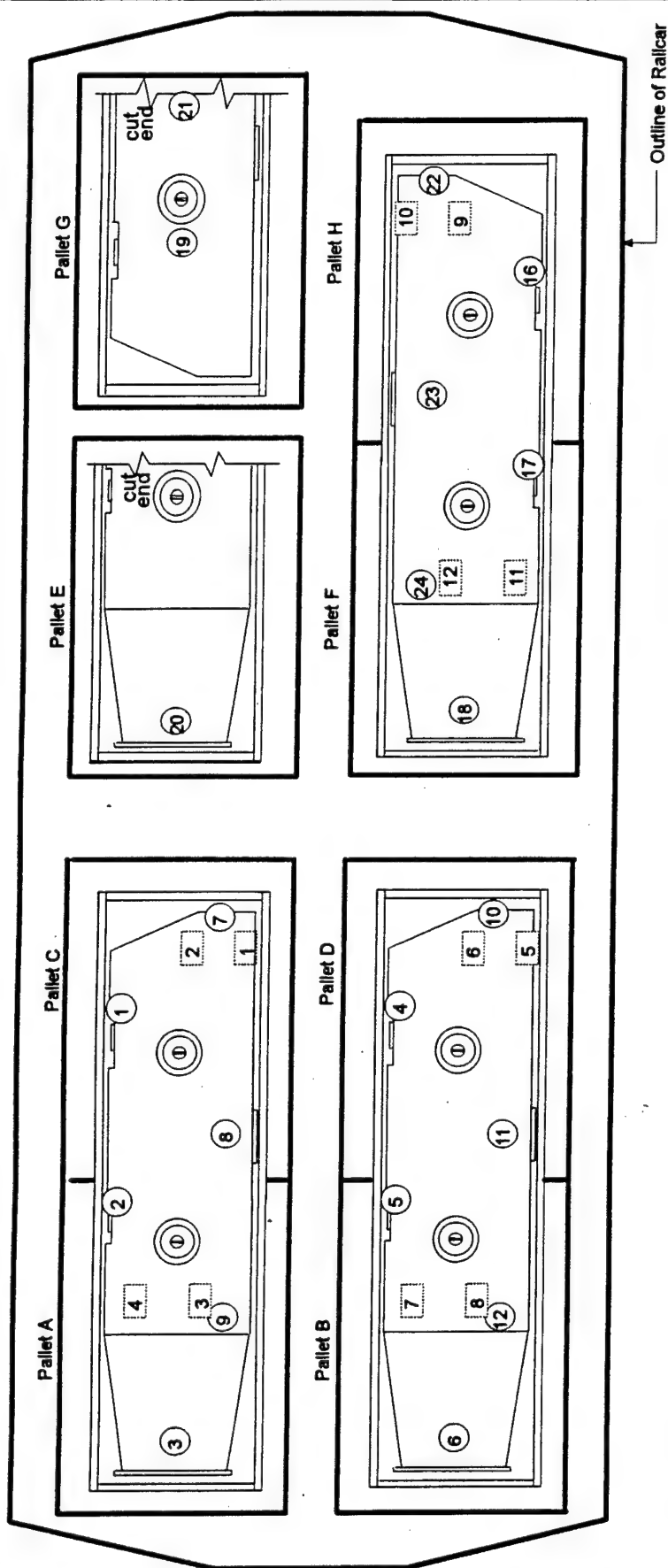
Figure 4-18 Test D - Baseline CEM Profile - 1 minute Intervals

Total Weight, 4 Mines = 2,860 lbs (1,297 kg)

MK 25 Ship Mines

(Unused mines - internals coated with hot-melt)

- 5 Thermocouple Number and Approximate Location
- 1 Sample Number and Approximate Location



See Figure D-1 for railcar placement in chamber
 See Figure D-2 for pallet placement on railcar
 See Figure D-8 for mine placement on pallets

(Not to Scale)

Figure 4-5 MK 25 Ship Mines Hot-Melt Coated Internals and Spiked with TNT

decontamination. The ship mines in Test 26 were spiked (2270 grams per mine) by Weston during the pilot study in 1989

Both Test 6 and 11 experienced power interruption and Test 6 underwent a safety shutdown (due to high chamber pressure) neither of which could be accounted for or explained. Adjustments in treatment time were made from Test 6 (48 hours) to Test 11 (32 hours).

During Test 26, a positive chamber pressure caused from flashing of the 5 pounds (2.3 kg) of TNT in the MK 25 Ship Mine resulted in a system safety shutdown. This portion of the test is now referred to as Test 26A.

Test 26A - As the ship mine temperatures reached 400°F (204°C) there were temperature peaks to 1100°F (593°C) possibly indicating thermal degradation of the explosive. Audible hisses were heard within the chamber and smoke was observed coming from the thermal oxidizer stack. Temperature and CEM peaks (Figures F-54 through F-57) continued over the next 45 minutes before beginning a gradual decline as the chamber was vented with fresh air. The system was shutdown and the chamber inspected. There was no visible damage to the chamber or any fixtures. No samples were taken. As discussed in the report, temperature spikes that resulted from thermal decomposition of residual amounts of explosives were observed often. In Test 26, this same phenomena is observed, however on a much larger scale. This test demonstrated that the HGD process is effective for removing residual explosives from projectiles or equipment, but that it could pose a safety problem if large quantities of explosives were present in a particular piece of equipment.

At the conclusion of this incident, there were no distortions or visible fractures to the ship mines being decontaminated. There were indications that molten hot melt had spewed from the fill plug when the contents flashed.

Test 26B - The system was restarted, with this test being a continuation of Test 26A, to complete decontamination of the ship mines and any residue remaining in the chamber. There appeared to be no additional thermal degradation of the explosive.

Most soot observed at the end of Test 26A was gone, however a reddish ash was observed in the ship mines closest to the diffuser and a black ash was visible in the ship mines nearest the door.

Another test (test conditions - 24 hours at 700°C, (371 °C) decontaminating ship mines previously spiked by Weston was originally included in the test plan but that test was removed from the schedule after the experiences of Test 26. Removal of that test was acceptable to TVA, HWAD, and USAEC (Environmental Technology Division) because residues remaining in typical washed out ship mines should be less than the 2270 grams of the Weston spiked ship mines. However, the current steamout/meltout process has not been demonstrated at HWAD to achieve contamination levels below the 5 pounds (2.3 kg) of TNT used in this test. From CEM data and analytical results of other tests treating hot melt items (Tests 6, 11, 19, 21, 25, and 26), it is likely decontamination of the TNT explosive and volatilization of the hot melt would be complete after 24 hours of treatment at 700°F (371 °C).

Ship Mine/Depth Bomb Wipe Samples

At treatment times of 48 hours (Test 6) and 32 hours (Tests 11 and 26) and a control temperature of 700°F (371 °C), all the ship mines were decontaminated to a DRE of 99.999 percent or greater. The average ship mine temperature ranged from 692 to 790°F (367 to 421 °C).

Chamber Wipe Samples

Explosive residues were detected in two of twenty-four samples with an average quantity of 8.5252E-06 mg/sq cm. This was similar to the surface area amounts detected in the ship mine wipe samples.

CEM Trends

As the ship mine temperatures reached 600°F (316°C) there were elevated CO readings for 16 hours in Test 6 before beginning a gradual decline. In Test 11 there were CO peaks and then elevated CO readings for 19 hours before beginning a gradual decline. This was likely due to the volatilization of the hot melt. During the incident recorded in Test 26A, the CEM readings of NO_x, CO, and total hydrocarbon were off scale at 500, 1000, and 100 ppm respectively, which indicated vigorous volatilization of the explosive. During this time period there were also extreme temperature spikes noted. Between the high CEM readings and thermal couple temperature spikes, it is likely that the explosives were thermally decomposing. These off scale peaks and extreme elevation in temperature readings did not occur in Test 26B. There were elevated CO and total hydrocarbon reading at the beginning of Test 26B as the MK 25 Ship Mine reached 600°F (316°C). These readings remained elevated for 8 to 12 hours before beginning a gradual decline. This data is an indication that volatilization of the hot melt would be complete after 24 hours of treatment at 700°F (371°C).

Gas Sampling

Analytical results of gas samples from the chamber exhaust duct indicated that explosives and thermal decomposition products from explosives were present. It was not possible to obtain a material balance between the amount of explosive present in the test items and the amount recovered in the resin tube. One reason for the discrepancy is that there appears to be many types of breakdown products present in the gas stream making it impossible to account for all the explosive material used in the system. Details are described in Section 3.4.1.

4.2.3 Explosive: Comp B

General Observations

Tests 3, 8, 13, 18, 23, and 27 were each conducted with 96 175mm projectiles in racks placed on a railcar. Each railcar contained 72 inert

projectiles for thermal mass and 24 contaminated projectiles which were internally monitored for metal temperature. The contaminated projectiles in the first four tests (Tests 3, 8, 13, and 18) were spiked with Comp B as described in Appendix B. The last two tests (Tests 23 and 27) were performed with projectiles which had been melted-out and contained residues of Comp B explosive. The projectiles in the first five tests (Tests 3, 13, 18, and 23) were arranged on the railcar generally as shown in Figure 4-6 and in Photo 4-2. The location of the contaminated projectiles in the adapters was varied in the five tests to provide temperature monitoring and sampling over as many locations as possible.

Based on the information gained from Test 26A and 26B, the last test (Test 27), designed for 175mm projectiles on railcars, was modified so that all 24 contaminated projectiles were located in the two racks nearest the diffusers as shown in Figure 4-7. This test was performed with this configuration to check for signs of chain reaction thermal degradation of projectiles contaminated with Comp B. Test 27 was the last test scheduled to be conducted on Comp B prior to the full chamber load tests that would represent production loads at HWAD. Based on information from previous tests, a special test was devised and conducted to make a smooth transition from railcar loads to a full chamber load. This test was designated as Test 96.

Test 96 was conducted on 96 melted out 175mm projectiles which contained residues of Comp B explosive. The test was performed as a preliminary test to a full-chamber load test with 480 projectiles. The effects of an increased quantity of explosive residue (4 times greater than previous tests) on system operation was studied. The increase in the quantity of Comp B in the chamber did not adversely affect the HGD system operation.

The temperature recording time was continued to produce a temperature profile at 15 second intervals instead of the 15 minute intervals previously

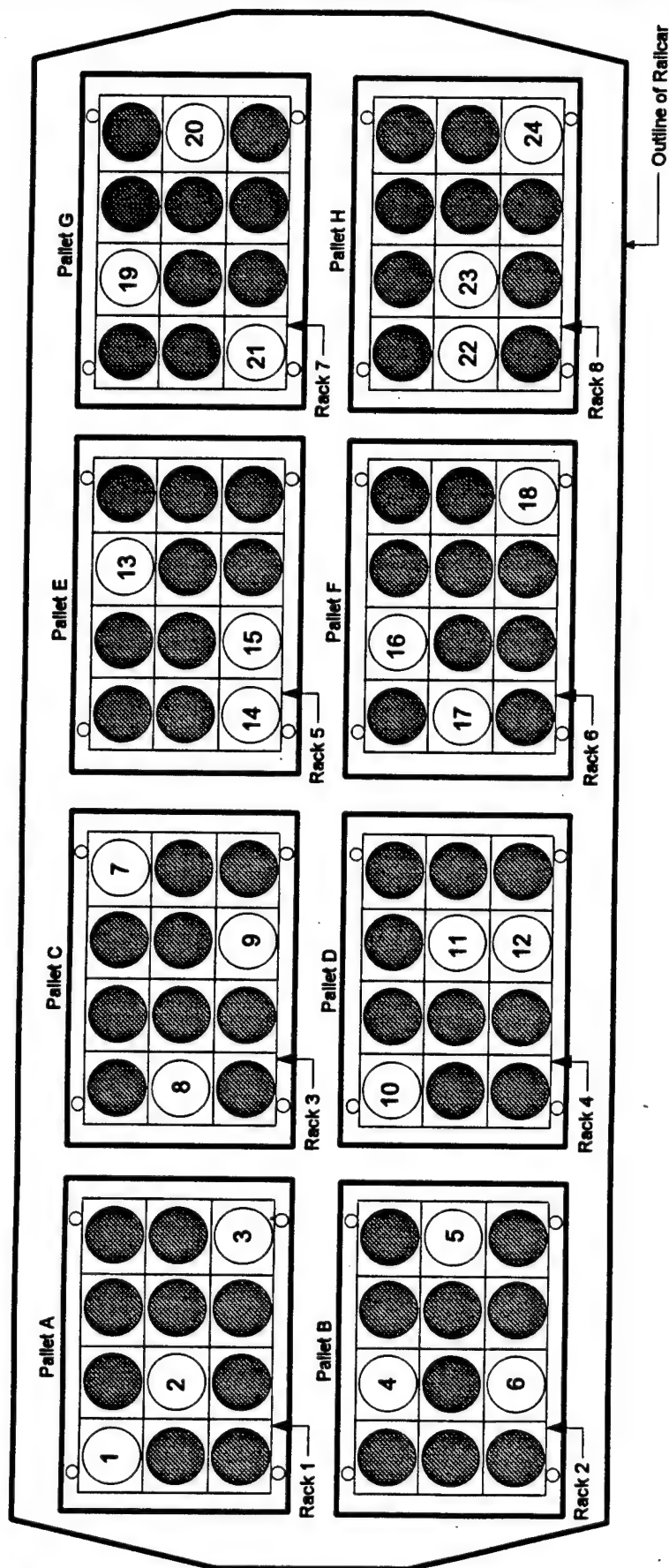
175mm Projectiles

(Items from FF-13)

Total Weight, 96 Projectiles = 11,040 lbs (5,008 kg)

7 Spiked projectile sampled

Inert projectile added for thermal mass



See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-6 for rack placement on pallets

(Not to Scale)

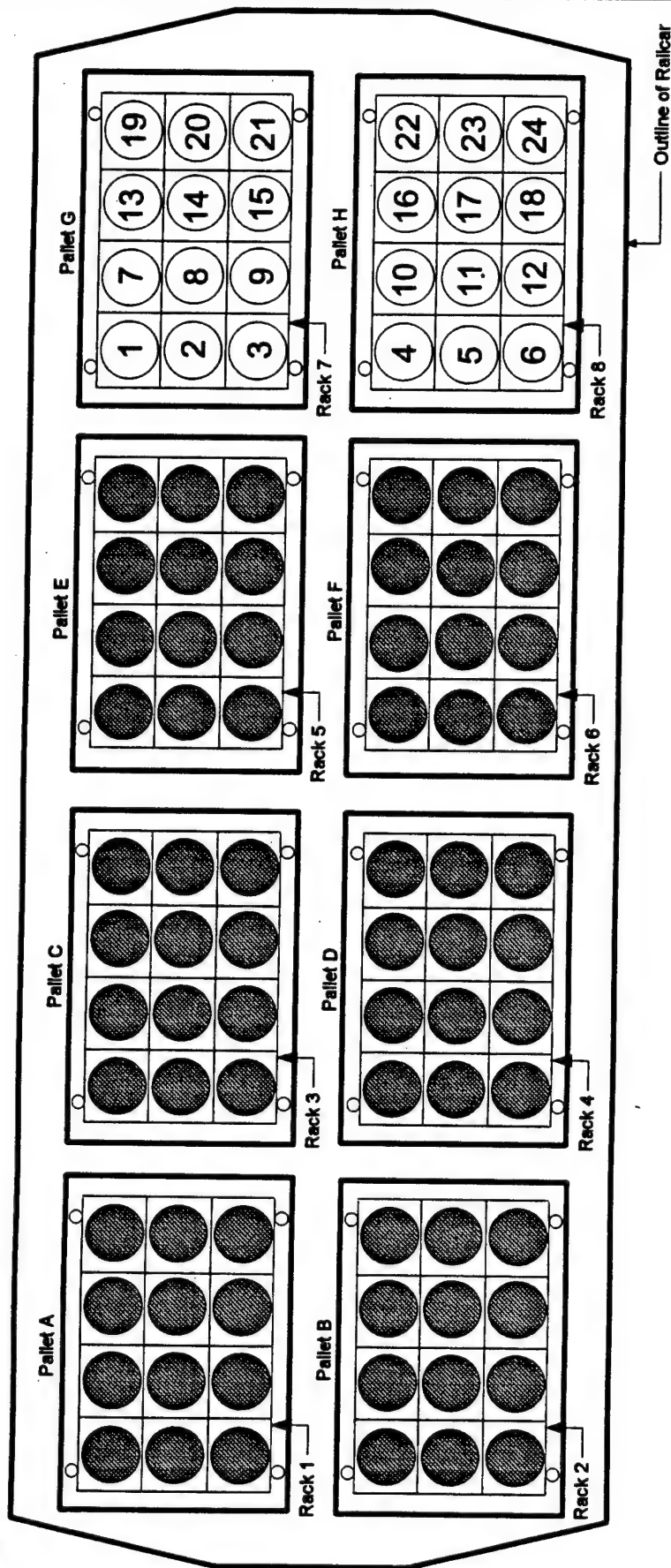
Figure 4-6 175mm Projectiles Spiked with Comp B

Total Weight, 96 Projectiles = 11,040 lbs (5,008 kg)

175mm Projectiles (Projectiles contained Comp B residue)

15 Contaminated projectile sampled

Inert projectile added for thermal mass



Test 27

(Not to Scale)

1 October 1984
6 Hours at 550 °F / 288 °C

See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-6 for rack placement on pallets

Figure 4-7 175mm Projectiles with Comp B Residue

used. This test confirmed prior test data that larger loads, such as 480 projectiles, could be decontaminated effectively. The partial chamber load configuration is shown in Figure 4-8 and in Photo 4-11.

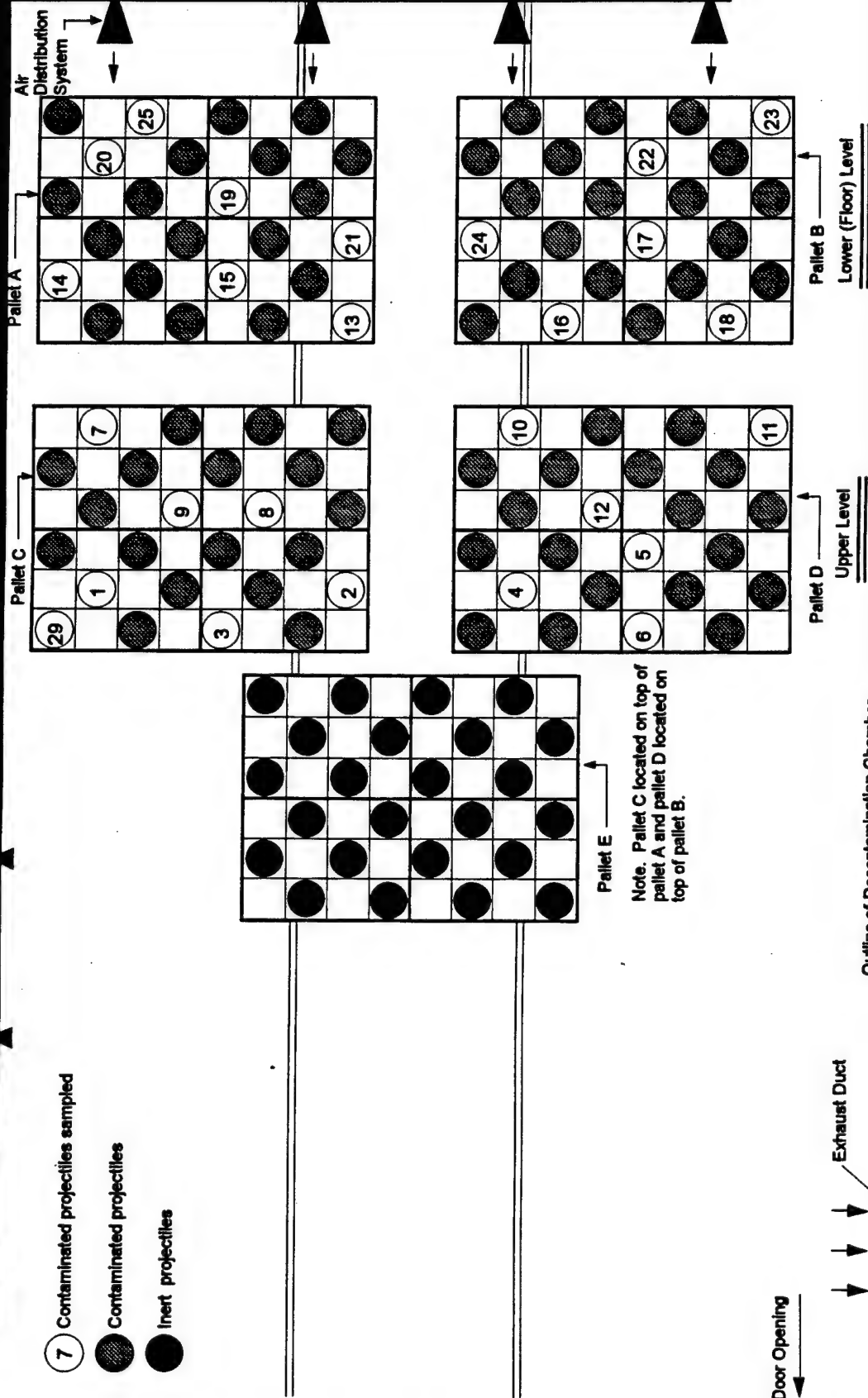
The decontamination process continued as tests 30 through 34 were conducted with 480 melted-out 175mm projectiles on pallets without the railcar. Twenty-four projectiles were palletized on a standard 40-inch by 48 inch (102 centimeter by 122 centimeters) metal pallet. Six 175mm projectiles were placed on a 5-inch adapter rack and 4 racks were banded together to form one pallet. The chamber at maximum capacity accommodated 20 pallets arranged two tiers high and two wide. Placement of the pallets in the chamber and temperature probe location is shown in Figures 4-9 and 4-9a and in Photo 4-3. Thirty-eight projectiles (32 TVA and 6 DZB probes) were monitored for temperature; from these thirty-eight projectiles the twenty-two TVA and two DZB probes with the lowest average steady state temperature were sampled. Probe locations were not intentionally varied from test to test but variations in arrangement of projectiles on pallets and pallets in the chamber increased the coverage of temperature and sampling locations. Four hundred eighty projectiles is considered a full chamber load for production purposes at HWAD. There was no evidence of a chain reaction caused by thermal degradation of Comp B. This process is an effective, safe, and economical method to decontaminate all explosive contaminated items tested during this program. Economic considerations are presented in Section 5.0.

Projectile Extraction Samples

Test 3 was conducted at a control temperature of 500°F (260°C) and a treatment time of 6 hours. Credible analytical results were not obtained because the samples fouled the HPLC column and obscured all response to explosive compounds. It was considered likely that new nose plugs used to close the projectiles during sample extraction contained a coating of Cosmoline or other preservative which contaminated the samples. To

Total Weight, 96 projectiles = 11,040 lbs (5,008 kg)

175mm Projectiles (Projectiles contained Comp B Residue)



Note. Pallet C located on top of pallet A and pallet D located on top of pallet B.

Figure 4-8 175mm Projectiles with Comp B Residue

Total Weight, 480 projectiles = 55,200 lbs (25,038 kg)

175mm Projectiles (Projectiles contained Comp B Residue)

- 6 Contaminated projectile sampled
- Contaminated projectiles
- E D/L Point #6
- F D/L Point #7
- PP Maples Process Probe

Air Distribution System
Pallet A

Pallet C

Pallet E

Pallet G

Pallet I

Pallet K

Pallet B

Pallet D

Pallet F

Pallet H

Pallet J

Lower (Floor) Level

Outline of Decontamination Chamber

Exhaust Duct

Door Opening

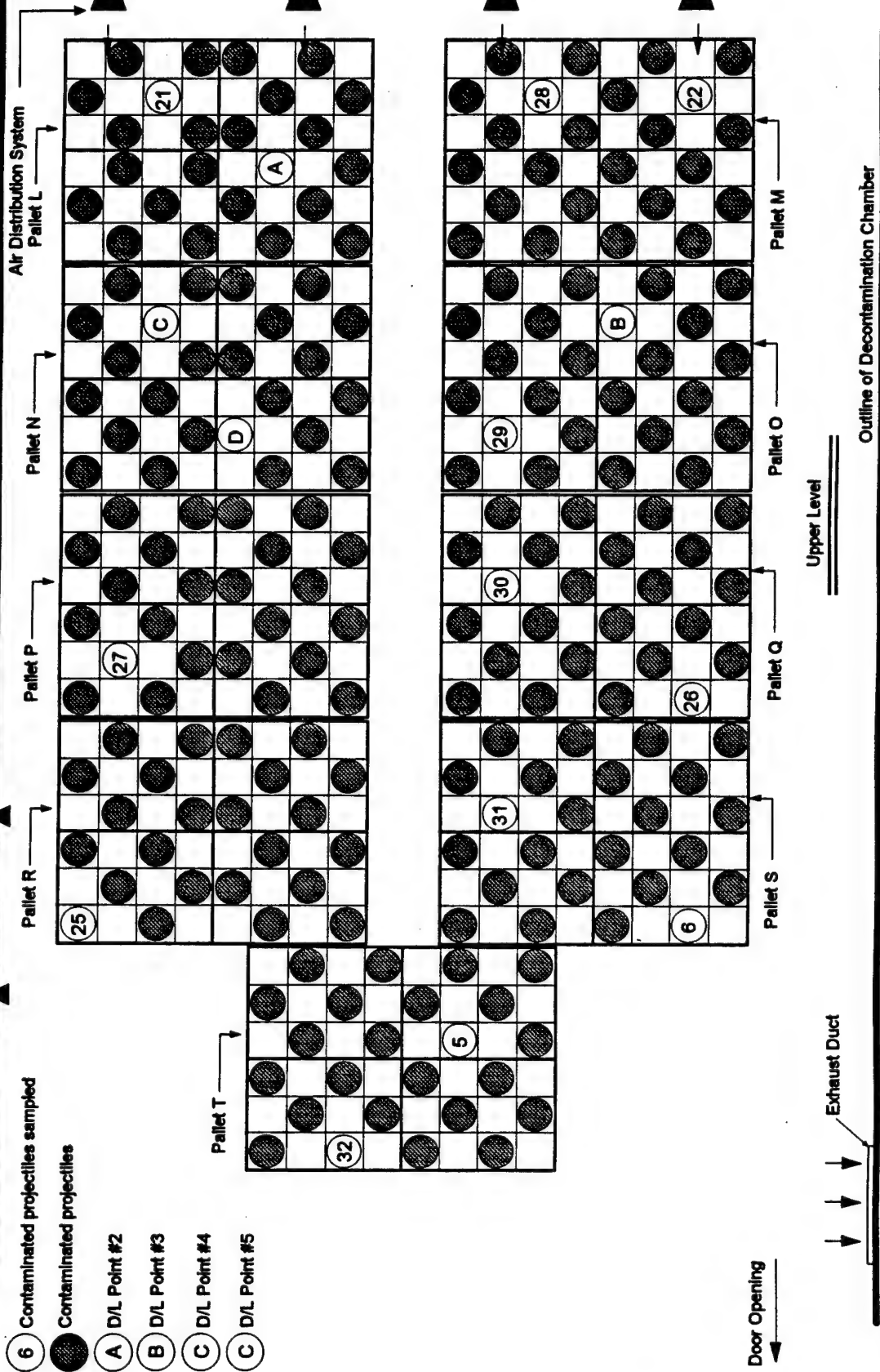
See Figure D-6a for 5-inch adaptor placement on pallet

(Not to Scale)

Figure 4-9 175mm Projectiles with Comp B Residue - Lower Level - Full Chamber Load

175mm Projectiles (Projectiles contained Comp B Residue)

- 6 Contaminated projectiles sampled
- Contaminated projectiles
- A D/L Point #2
- B D/L Point #3
- C D/L Point #4
- C D/L Point #5



See Figure D-6a for 5-inch adaptor placement on pallet

(Not to Scale)

Figure 4-9a 175mm Projectiles with Comp B Residue - Upper Level - Full Chamber Load

prevent this contamination in the tests that followed, all plugs were flashed in the flashing furnace (FF-13). As in Test 1 with TNT, this procedure eliminated the source of contamination.

At treatment times of 6 hours and control temperatures of 550°F (288°C) (Tests 8, 13, 18, 23, and 27) there was decontamination to a DRE of 99.999 percent or better for the 24 sampled projectiles in each test. The average projectile temperature for the 6 hours ranged from 542 to 650°F (283 to 343°C).

For Test 96 the treatment time of 2 hours and the treatment temperature of 550°F continued and resulted in decontamination to a DRE of 99.999 percent or greater. A full set of projectile samples were not taken; only two randomly selected projectiles were extracted. The average projectile temperature for the two hours ranged from 610 to 795°F (321 to 424°C). Tests 30, 31, 32, 33, and 34 were also conducted at a control temperature of 550°F (288°C) and a treatment time of 6 hours. All sampled projectiles were decontaminated to a DRE of 99.999 percent or better. The average projectile temperature during the 6 hours ranged from 554 to 846°F (290 to 452°C).

The overall results from these tests and extraction analyses indicate that regardless of the "load" in the chamber, decontamination to a DRE of 99.999 percent or better will occur.

Random testing of 10-percent (200 out of 2000) of the decontaminated projectiles using Webster's Reagent was conducted by DZB. The test results were all negative.

Chamber Wipe Samples

During Tests 8, 13, 18, 23, and 27 explosive residues were detected in five out of forty samples (five tests, eight samples per test) with three of the

residues located at the "rail" sample location (Figure 4-1). Since the rail was still connected (thermally) to the outside of the chamber it acted as a heat sink and allowed the residue to collect at that point. Also, walking into and out of the chamber, and driving equipment into and out of the chamber could have been the source of contamination. Even when there were explosive residues detected there was no indication of a build up in the system. The quantity of residue averaged $2.0297\text{E-}05$ mg/sq cm, which was similar to the surface area residue levels of the above projectile extraction samples. Wipe samples were not collected during Test 96.

Chamber wipe samples were collected and analyzed for the five full load tests (Test 30, 31, 32, 33, and 34). Explosive residues were detected in thirteen of forty samples (five tests, eight samples per test) with four of the residues located at the "rail" sample location. Again, the rail probably acted as a heat sink and contributed to the residue being collected at this point. The residue quantity averaged $1.7981\text{E-}05$ mg/sq cm, which was similar to the surface area residue levels detected in the projectile extraction samples.

CEM Trends

CEM data was not collected for Test 3.

In Tests 8, 13, 18 and 23 small peaks were noted on the CEM for total hydrocarbon and medium to large peaks for NO_x and CO as the projectile temperatures approached 400°F (204°C). During steady-state operation the CEM data generally followed the baseline conditions observed during tests without explosives. Small increases and decreases in CEM data occurred as the temperature would rise and fall. In Test 18 a power interruption caused a sudden change in CEM data.

During Test 27 the NO_x and CO peaks correspond with the peaks shown on the temperature profile. The peaks are much better defined in the CEM 1-minute intervals and temperature 15-second intervals. The change in

temperature caused by the upset in the temperature controller was also reflected in the CEM profile.

With the increase in the number of projectiles in the chamber for Test 96, there was not a significant increase in the levels of NO_x and CO. The THC analyzer was not operational during this test. As before, the NO_x and CO peaks correspond with the peaks recorded on the temperature profile. The temperature peaks indicate the Comp B being thermally degraded.

In the full chamber load tests (Tests 30, 31, 32, 33, and 34) peaks were noted on CEM for total hydrocarbons, NO_x, and CO similar to what was observed in the railcar tests above but of larger magnitude. The estimated amount of explosive in a chamber load is 9600 grams as compared to 480 grams in a railcar arrangement. The THC, NO_x, and CO peaks occurred during the period of temperature peaks.

Generally, for all 5 full chamber load tests the THC, NO_x, and CO peaks, NO_x in particular, increased from 50-100 ppm and in Test 27 to 200-300 ppm. These peaks occurred during the heat-up stage of each test, usually as the temperature approached 400°F (204°C). These peaks indicated on the CEM profile correspond with the temperature where Comp B "flashes" or thermally degrades. After steady state conditions are achieved, the NO_x levels reduce to the 20 to 30 ppm range as reported earlier as baseline.

Unusually high, momentary peaks in CEM data is almost always associated with unstable conditions as noted on the CEM profile figures, i.e., power interruptions, safety shutdowns, or programmed instrument recalibrations.

175mm Projectile Paint Sample

Samples of paint were scraped from the outside metal surface of untreated 175mm projectiles on September 2, 1994. The paint was analyzed for metals with an Inductively-Coupled Plasma (ICP) Emission Spectrometer. Laboratory results are given in Table 4-1.

Table 4-1
175mm Projectile Paint Sample

<u>Element</u>	<u>Concentration, mg/kg</u>
Ag	<0.2 (<MDL)
Al*	9000
As	1.73
Ba	576
Be	<0.3 (<MDL)
Cd	21.2
Cr	3310
Hg	<0.3 (<MDL)
Ni	101
Pb	12600
Se	<0.37 (<MDL)
Sb	47.2
Tl	1020

*Semi-quantitative number

It appears that the metals in the paint were oxidized during system operation and carried out, along with the explosive residue vapors, in the exhaust gases from the chamber. The paint turned from a green color before a test to a red color after the test. USACHPPM's exhaust gas sampling showed emissions of metals from the thermal exhaust stack. Results of their sampling is shown in Section 4.2.3, Table 4-2.

Gas Sampling

During Tests 31 through 34 USACHPPM conducted atmospheric emissions sampling of the thermal oxidizer exhaust stack. Samples for total hydrocarbons (THC), NO_x, CO, SO₂, and particulates were taken during

Table 4-2
Samples for Total Hydrocarbons, NO_x, CO, SO₂, Particulates,
and Metals During Full Chamber Load Tests

	Emission <u>Rate, tons/yr*</u>	Emission <u>Rate, gr/hr</u>
THC	2.3 E-02	
NO _x	1.631E+01	
CO	6.4 E-01	
SO ₂	1.062E+01	
Particulates	1.238E+00	
Metals		
Ag		4.94 E-03
As		3.50 E-03
Ba		2.994 E-02
Be		3.1 E-04
Cd		2.829 E-02
Cr		1.4378E-01
Ni		2.2324E-01
Pb		6.069 E-02
Sb		1.19 E-03
Se		6.3 E-04
Ti		4.3 E-04

* Based on 8760 hours of operation/year

heatup of the projectiles when thermal degradation of the explosive was observed. Samples for metals were taken near the end of steady state operation when the projectiles were at maximum temperature. A copy of the USACHPPM report is included in Appendix G. An average of the results is shown in Table 4-2. Analytical results of gas samples from the

chamber exhaust duct indicated that explosives were present. It was not possible to obtain a material balance between the amount of explosive present in the test items and the amount recovered in the resin tube. One reason for the discrepancy is that there appears to be many types of breakdown products present in the gas stream making it impossible to account for all the explosive material used in the system. Details are described in Section 3.4.1

Projectile Temperatures

Projectile temperature data was not reported for Test 3.

In Test 8, 13, 18, and 23 projectile temperatures were recorded on 15 minute intervals. Temperature profiles of all test are reported in Appendix F. Temperatures were monitored in as many different locations as possible.

In Test 27 the projectile temperatures were recorded every 15 seconds instead of the 15 minute intervals of tests prior to Test 26. The interval was decreased to measure the rapid increase in projectile temperatures that was due to thermal degradation of the explosive. Temperature peaks were observed during the time period when the projectiles neared 400°F (204°C), with short term temperature increases (exothermic reaction) of up to 350°F (194°C) for the selected projectiles. There was no indication from the projectile temperature profiles that a chain reaction of flashing from one projectile to another was occurring.

In Test 96 the projectile temperatures were recorded every 15 seconds. The temperature peaks for the monitored projectiles are evident in the 15 second temperature profile presented in Appendix F. These individual elevated temperature "spikes" are hardly noticeable on the 15 minute profile. These peaks are also easily correlated to the CEM profiles during the same time frame. The higher temperature peaks were observed during the

time period when the projectiles neared 400°F (204°C), with temperature increases of up to 750°F (417°C) for the selected projectiles.

The momentary (short-term) elevated temperature from these tests or from the full chamber load tests did not effect the operational condition of the facility or HGD system.

In the five full chamber load tests the projectile temperatures were recorded every 15 seconds. Short-term temperature peaks were observed during the time period when the projectiles neared 400°F (204°C), with temperature increases of up to 500°F (278C) for the selected projectiles.

The average projectile temperature during the 6 hours of steady-state operation of each of the five tests ranged from 554 to 846°F (290 to 452°C).

4.2.4 Test B2

General Observations

Due to the presence of substantial residues of TNT and RDX reported from Test 1, this unscheduled test was inserted into the Test Sequence (Table 2-2) after Test 3 and was conducted to decontaminate the railcar used in Test 1. The sand bed and metal racks were removed from the railcar. Test conditions were 6 hours at 550°F (288°C).

Explosive residues of 3.7993E-05 mg/sq cm were detected on the railcar prior to the test. These analytical results indicated that the railcar was one of the sources of contamination. No residues were detected after the test indicating the railcar was decontaminated and would not be a source of cross-contamination during future tests.

There were no projectile extraction samples required for this test.

Chamber Wipe Samples

Explosive residues of 8.8826E-06 mg/sq cm were detected. Two of five insulation samples taken from gaps in the wall seams within the chamber tested positive for RDX and TNT.

CEM Trends

No increased CEM activity which would be attributable to explosives was noted.

4.2.5 Tests B3 and B4

General Observations

These two unscheduled tests were conducted on an empty chamber to decontaminate the chamber of any explosive residue remaining after Tests 1 and 3. Residues of TNT and RDX were detected in samples from Test 1. Accumulation of residues in the chamber during routine operation could present a hazard to personnel or the facility. Test B4 was begun immediately after chamber wipe samples were taken from Test B3. Test conditions for Test B3 were 6 hours at 550°F (288°C) and for Test B4, they were 24 hours at 700°F (371°C).

Also for Test B3 special samples were taken from area soil, insulation from within the chamber, inert and flashed projectiles and nose plugs. These samples were analyzed to determine the source(s) of contamination. The analytical results were that the soil, insulation, and untreated flashed projectiles did not contain detectable explosive residues. The untreated inert projectiles and projectile nose plugs did contain explosive residues. These results are presented in Table F-6.

During Test B4 adjustments were made to the Maples controller to improve operating performance. The changes discussed in Appendix F improved the system operation.

There were no projectile extraction samples required for these tests.

Chamber Wipe Samples

Test B3 - Explosive residues were detected in one of eight samples; the amount of residue was 4.9373E-06 mg/sq cm.

Test B4 - Explosive residues were detected in one of eight samples with the amount of residue being 1.1036E-05 mg/sq cm.

CEM Trends

CEM data was not collected during these tests.

4.2.6 Explosive: HBX

4.2.6.1 3-Inch Projectiles

General Observations

HBX explosive was decontaminated in Tests 5, 9, and 14. The tests were conducted with 192 3-inch projectiles in racks placed on a railcar. Each railcar contained 168 inert projectiles for thermal mass and 24 spiked (described in Appendix C) projectiles which were internally monitored for metal temperature. They were arranged on the railcar generally as shown in Figure 4-10 and in Photo 4.4. The locations of the spiked projectiles in the racks were varied in the three tests to provide temperature monitoring and sampling over as many locations on the railcar as possible.

Based on the results from Test 5, it was necessary to increase the temperature for Test 9 from 550°F (288°C) to 600°F (316°C). The optimum temperature for decontaminating HBX in 3-inch projectiles appeared to be 600°F.

Projectile Extraction Samples

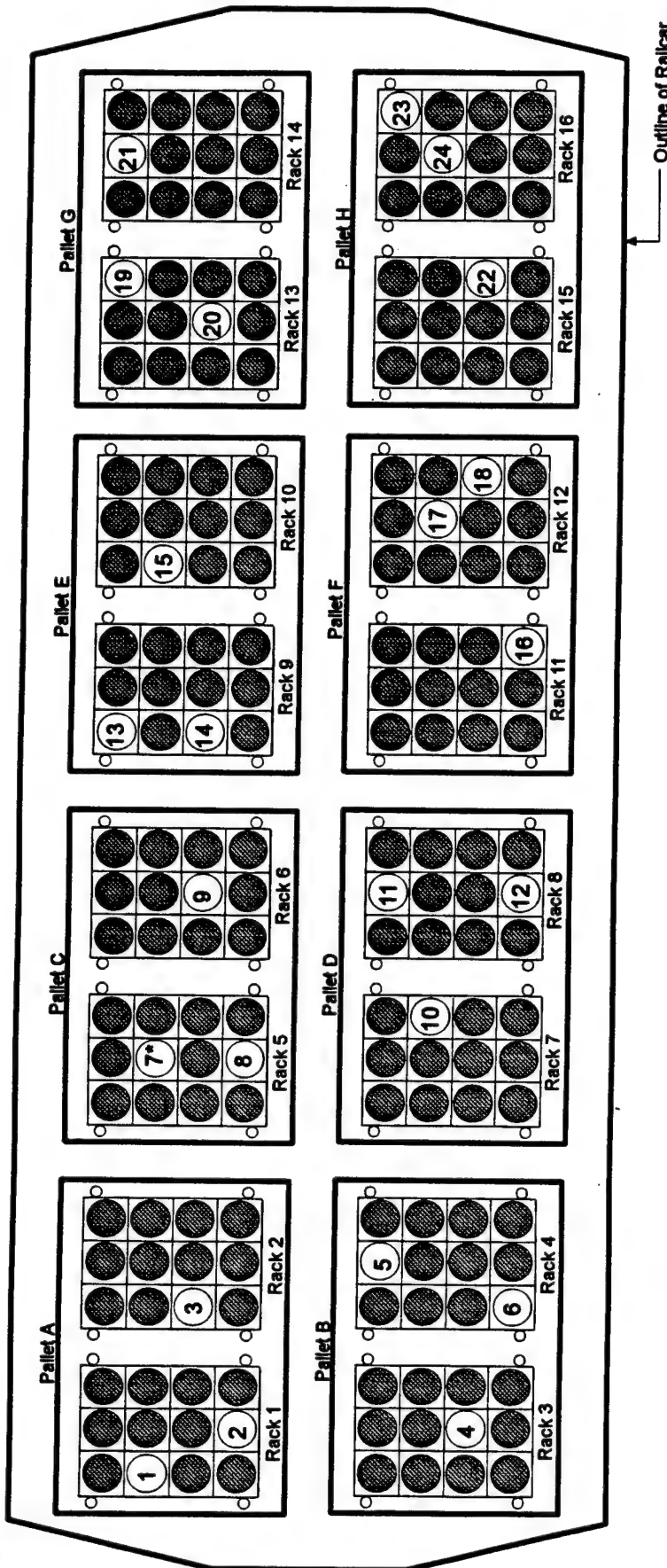
At a control temperature of 550°F (288°C) and a treatment time of 6 hours (Test 5), there was decontamination to a DRE of 99.979 percent or better.

Total Weight , 192 projectiles = 1,728 lbs (784 kg)

3-inch Projectiles

(Items from FF-13)

- 5 Spiked 3-inch projectile sampled
- Inert 3-inch projectile added for thermal mass



Door Opening

Outline of Railcar

(Not to Scale)

See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-3 for rack placement on pallets

Figure 4-10 3-Inch Projectiles Spiked with HBX

The average projectile temperature during the 6 hours ranged from 557 to 591°F (292 to 311°C).

At control temperatures of 600°F and treatment times of 6 hours (Tests 9 and 14) all sampled projectiles were decontaminated to a DRE of 99.999 percent or better. The average quantity of residue in the detectable samples was 2.956E-06 mg/sq cm. The average projectile temperature ranged from 594 to 671°F (312 to 355°C).

Chamber Wipe Samples

Explosive residues were detected in five of twenty-four samples with an average quantity of 7.2031E-06 mg/sq cm. This amount was similar to the surface area residue levels of the projectile extraction samples.

Of the five samples, two were on the rail which apparently serves as a heat sink and allows collection of residue. Two samples were in the elbow of the duct to the oxidizer, and the other contaminated sample was located on the ID fan blade. The amount of residue measured does not present a safety hazard to either personnel or HGD system. These samples do not indicate an accumulation of explosive residue within the chamber.

The fact that only very small quantities ($\mu\text{g}/\text{cm}^2$) of explosives were found in the chamber suggested that there was no buildup of explosives in the system. This was also supported by the fact that wipe samples were taken from different locations to determine the buildup rate between several tests. Since no accumulation was observed, it was determined that the system operation did not and would not present an explosive hazard to personnel and the facility during routine operation.

CEM Trends

In each of the tests of 3-inch projectiles contaminated with HBX, the CEM profiles indicate normal or background levels of NO_x , CO, and THC after the

system reaches temperature and goes into steady-state operation. In all three tests, there were NO_x peaks from 40 to 50 ppm as the projectile temperatures neared 400°F (204°C). CO peaked at less than 10 ppm and THC data indicated no significant change during the three tests.

Gas Sampling

Analytical results of gas samples from the chamber exhaust duct indicated that explosives and thermal decomposition products from explosives were present. It was not possible to obtain a material balance between the amount of explosive present in the test items and the amount recovered in the resin tube. One reason for the discrepancy is that there appears to be many types of breakdown products present in the gas stream making it impossible to account for all the explosive material used in the system. Details are described in Section 3.4.1.

4.2.6.2 MK 54 Depth Bombs

General Observations

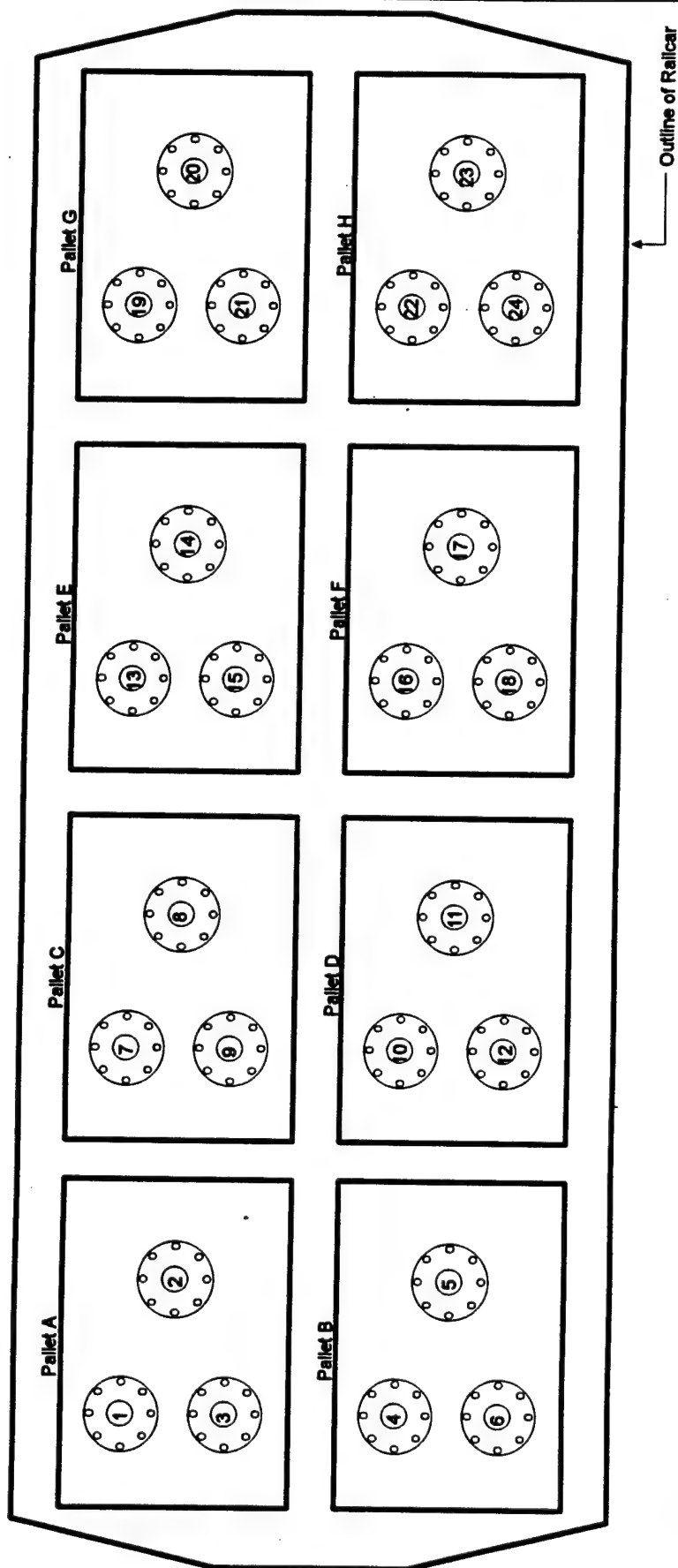
Residues of HBX explosive were decontaminated in Tests 16, 19, 21 and 25. The tests were conducted with MK 54 Depth Bombs (sawed ends) on racks placed on a railcar. Each railcar contained 24 demilitarized depth bomb ends arranged as shown in Figure 4-11 and in Photo 4.5. The depth bomb ends were monitored for temperature and the arrangement was the same for all four tests.

Naval munitions are internally coated with a tar-like asphaltic material known as hot melt or flintcoat (Photo E-10). Tests indicate that explosives are soluble in the hot melt as it is melted during heatup of the chamber. It appears that the hot melt will retain an appreciable explosive content throughout a treatment cycle that would decontaminate explosive from a metal surface. Therefore in order to ensure decontamination of items coated with hot melt, the hot melt will have to be removed along with the explosive. In small-scale laboratory tests a treatment of 700°F (371°C) for

Total Weight, 24 pieces = 384 lbs (174 kg)

MK 54 Depth Bombs (Sawed ends contained HBX residue)

① Contaminated pieces sampled



Door Opening

Outline of Railcar

(Not to Scale)

See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-7 for sawed ends placement on pallets

Figure 4-11 MK 54 Depth Bombs (Sawed Ends) with HBX Residue

32 hours was required to volatilize 99 percent of the hot melt, leaving only an ashy residue which was suitable for chemical analysis.

Generally, the sampling indicated no buildup residue of hot melt or HBX was noted on the treated depth bombs. Usually after decontamination all depth bombs looked clean (visual inspection) and were covered to some extent with a dusty, ashy powder. The color often varied from gray to black to red. Very small quantities of explosive material ($1\text{E-}06$ mg/sq cm) were detected in the powdery residues.

Depth Bomb Wipe Samples

At a treatment time of 32 hours (Test 16) and a control temperature of 700°C (371°C), there was explosive residue ($3.762\text{E-}06$ mg/sq cm) detected in only one of twenty-four depth bomb ends. The average depth bomb temperatures during the 32 hours ranged from 712 to 749°C (378 to 398°C).

At treatment times of 24 hours (Tests 19, 21, and 25) and a control temperature of 700°F (371°C), there was explosive residues detected in twenty of the seventy-two depth bomb ends. The average quantity (range, $4.5267\text{E-}08$ to $3.1980\text{E-}05$ mg/sq cm) for the twenty detectable depth bomb ends was $6.2837\text{E-}06$ mg/sq cm. The average depth bomb temperatures ranged from 710 to 759°F (377 to 404°C).

The quantity of residues detected in these two sets of tests were similar to quantity of residues detected in the spiked projectiles (Tests 5, 9, and 14) that were decontaminated to a DRE of 99.999 percent or greater. As a result, it appears that if the hot melt is completely volatilized, then the explosive residue concentration will be negligible.

Chamber Wipe Samples

Explosive residues were detected in eight of thirty-two samples with an average quantity of 8.3272E-06 mg/sq cm. This was similar to the surface area amounts detected in the depth bomb wipe samples. It was assumed again, as in the depth bomb wipe samples, that if the hot melt is completely volatilized then the explosive residue will not be present in sufficient quantity to produce a safety hazard. The quantity of residue detected equates to the spiked projectiles in Tests 5, 9, and 14 which decontaminated to a DRE of 99.999 percent or greater which indicates there is no accumulation of explosive residue within the chamber.

CEM Trends

In each of the tests (Test 16, 19, 21, and 25), as the depth bomb ends temperatures approached 400°F (204°C) there were peaks on the CEM for total hydrocarbons, CO, and NO_x. This was likely due to the thermal degradation of the explosive.

As depth bomb temperatures neared 700°F (371°C) there were elevated CO readings on the CEM for 11 hours in Tests 16 and 19, 4 hours in Tests 21 and 25, then a gradual decline. This was likely due to the volatilization of the hot-melt.

Any changes in temperature such as at 0730 hours on 22 August 1994 during Test 16 [approximate 35°F (19°C) rise] there was a corresponding rise in CO levels on the CEM profile. These similar conditions held on the other tests (Test 19, 21, and 25) conducted on HBX contaminated MK 54 Depth Bomb ends.

4.2.7 Explosive: RDX

General Observations

RDX explosive was decontaminated in Tests 2, 7, 12, and 17. RDX was used as a substitute for Comp A-3 in spiked tests because no solvent or

miscible combination of solvents identified produced a homogeneous solution of Comp A-3 suitable for spiking. Comp A-3 explosive was decontaminated in Tests 22 and 28. Results from these tests are discussed in Section 4.2.11.

Comp A-3 is comprised of 91 percent RDX and 9 percent of a wax stabilizer. This was considered an acceptable substitution by TVA and USAEC (Environmental Technology Division). The tests were conducted with 96 each 3-inch and 5-inch projectiles in racks placed on a railcar. The projectiles were arranged on the railcar generally as shown in Figure 4-12 and in Photo 4.6. The location of the spiked projectiles in the racks was varied in the four tests to provide temperature monitoring and sampling over as many locations on the railcar as possible. Twelve each of the 3-inch and 5-inch projectiles were spiked with RDX as described in Appendix C.

The first test conducted with RDX was Test 2. The 3-inch projectiles heated more quickly than the 5-inch which caused the control thermocouple to be located in a 5-inch projectile.

Projectile Extraction Samples

At a control temperature of 500°F (260°C) and a treatment time of 6 hours (Test 2), there was decontamination to a DRE of 99.988 percent or better. The average projectile temperature for the 6 hours ranged from 521 to 546°F (272 to 286°C) for the 3-inch projectiles and from 509 to 555°F (265 to 291°C) for the 5-inch projectiles.

At control temperatures of 550°F (288°C) and treatment times of 6 hours (Tests 7, 12, and 17) 65 of the 66 sampled projectiles were decontaminated to a DRE of 99.999 percent or better. The other projectile was decontaminated to a DRE of 99.997 percent. The average 3-inch projectile temperature ranged from 537 to 621°F (281 to 327°C) and the 5-inch ranged from 549 to 612°F (287 to 322°C).

Total Weight, 192 projectiles = 7,298 lbs (3,309 kg)

3-inch/5-inch Projectiles

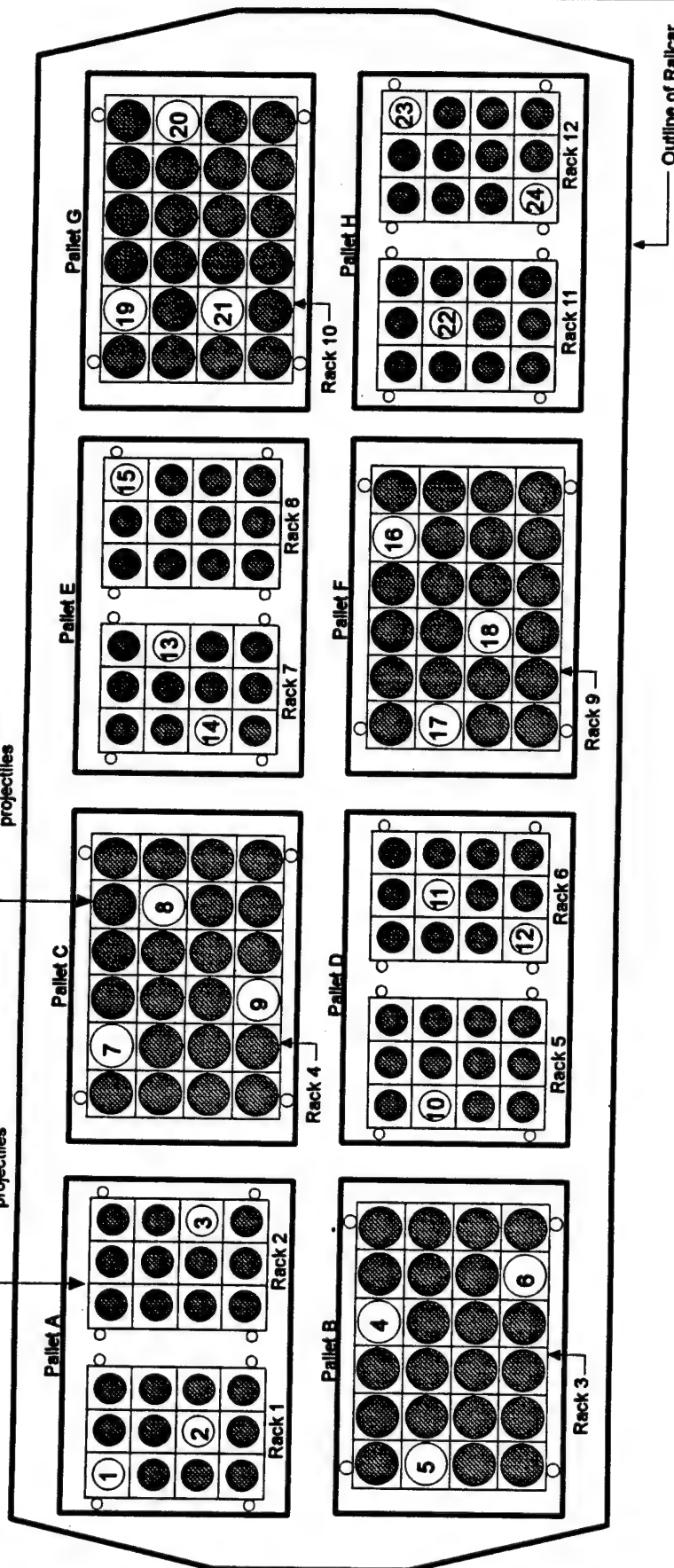
(Items from FF-13)

8 Spiked projectile sampled

Inert projectile added for thermal mass

Rack with 5-Inch projectiles

Rack with 3-Inch projectiles



(Not to Scale)

See Figure D-1 for railcar placement in chamber
 See Figure D-2 for pallet placement on railcar
 See Figure D-3 for 3-Inch rack placement on pallets
 See Figure D-4 for 5-Inch rack placement on pallets

Figure 4-12 3-inch / 5-Inch Projectiles Spiked with RDX

Chamber Wipe Samples

Explosive residues were detected in four of thirty-two samples (8 samples per test) but none in the same sample location. The quantity of residue averaged $6.565\text{E-}06$ mg/sq cm and was similar to the surface area amounts detected in the projectile extraction samples.

The samples containing residue occurred in Tests 2 and 7 in four separate locations. One location was the rail in the floor and another was in the floor trench indicating contamination could have been "tracked" into the chamber when unloading the test items. Still at the concentration noted above the equivalent amount extracted from the projectiles had DRE results of 99.999 percent or better.

CEM Trends

As projectile temperatures neared 400°F (204°C) there was slightly increased activity of CO and NO_x observed, although the activity was not as well defined as that noted with other explosives.

All tests show the CEM data changing with corresponding changes in the temperature profile which is to be expected but there were no significant changes in the CEM values during steady-state operation.

In Test 2 the dips in NO_x correspond with the drops in temperature which indicates a shift in NO_x due to additional outside air being drawn into and cooling the chamber.

In Test 7 CEM data indicates the NO_x monitor was recalibrated just as the system went into steady state.

Other tests show fluctuations in the CEM profile where power interruptions occurred.

Gas Sampling

Analytical results of gas samples from the chamber exhaust duct indicated that explosives and thermal decomposition products from explosives were present. It was not possible to obtain a material balance between the amount of explosive present in the test items and the amount recovered in the resin tube. One reason for the discrepancy is that there appears to be many types of breakdown products present in the gas stream making it impossible to account for all the explosive material used in the system. Details are described in Section 3.4.1.

4.2.8 Explosive: Yellow D (Ammonium Picrate)

General Observations

Yellow D (Ammonium Picrate) explosive was decontaminated in Tests 10, 15, 20, 24, and 29. The tests were conducted with 96 each 3-inch and 5-inch projectiles in racks placed on a railcar. The projectiles were arranged on the railcar generally as shown in Figure 4-13 and in Photo 4.10. The location of the spiked projectiles in the racks was varied in the five tests to provide temperature monitoring and sampling over as many locations on the railcar as possible. Twelve each of the 3-inch and 5-inch projectiles were spiked with Yellow D as described in Appendix C.

In Test 29 the projectile temperatures were recorded every 15 seconds instead of the 15 minute intervals previously recorded. The interval was decreased to measure the rapid increase in projectile temperatures that was due to thermal degradation of the explosive. Temperature peaks were observed during the time period when the projectiles neared 500°F (260°C), with temperature increases (exothermic reaction) of up to 200°F(111°C) for the selected projectiles.

Projectile Extraction Samples

At a control temperature of 600°F (316°C) and treatment time of 12 hours (Test 10), 6 hours (Test 15), 8 hours (Test 20), and again at 6 hours (Test

Total Weight, 192 projectiles = 7,296 lbs (3,309 kg)

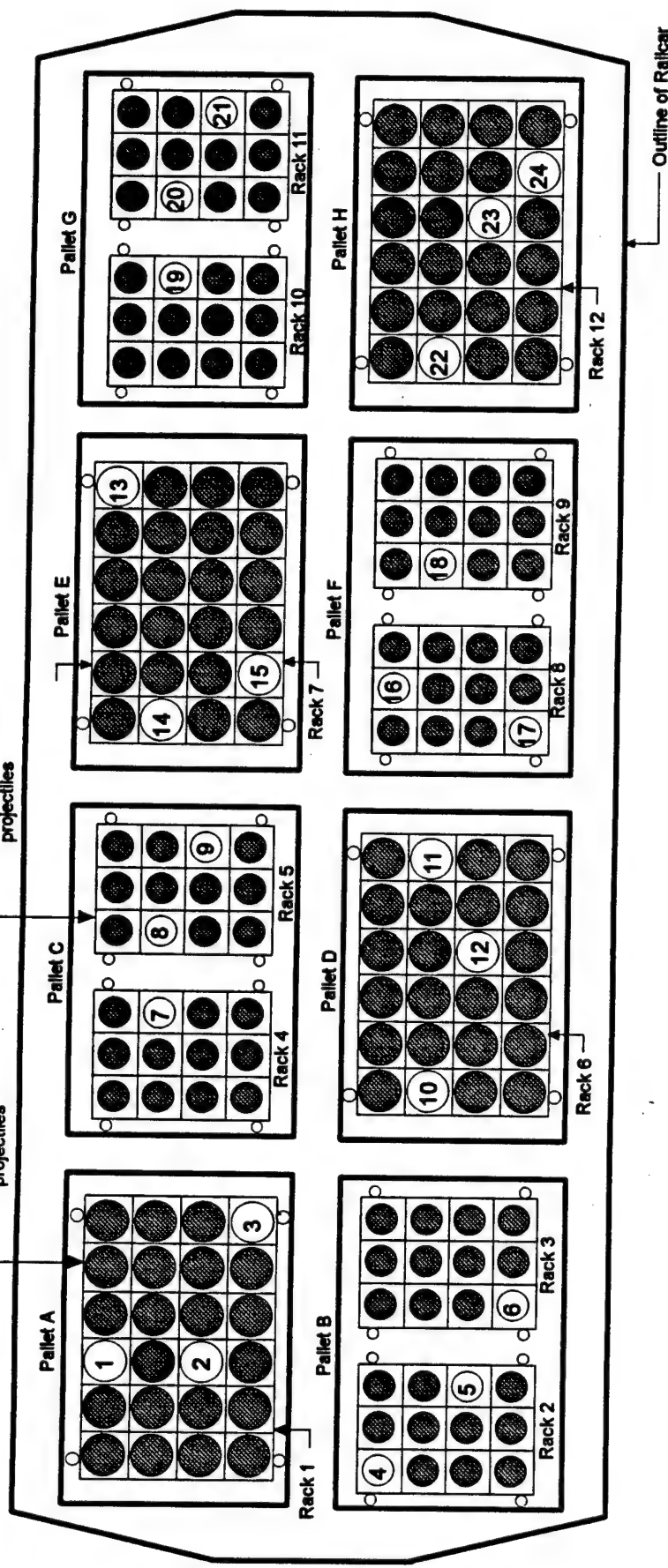
3-inch / 5-inch Projectiles (Items from FF-13)

9 Spiked projectile sampled

Inert projectile added for thermal mass

Rack with 5-inch projectiles

Rack with 3-inch projectiles



Door Opening

Outline of Railcar

(Not to Scale)

See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-3 for 3-inch rack placement on pallets
See Figure D-4 for 5-inch rack placement on pallets

Figure 4-13 3-inch / 5-inch Projectiles Spiked with Yellow D

24 and 29), all the projectiles were decontaminated to a DOE of 99.999 percent or greater. The average projectile temperature ranged from 589 to 673°F (309 to 356°C) for the 3-inch projectiles and from 593 to 692°F (312 to 367°C) for the 5-inch projectiles.

For Test 15 the treatment time was reduced to 6 hours and all the projectiles were decontaminated to a DRE of 99.999 percent or greater with respect to ammonium picrate, picric acid, and its other salts. There was however, HPLC chromatogram peaks indicating other unidentified compounds in the samples that were not present in Test 10 (treatment time-12 hours).

For Test 20 the treatment time was increased to 8 hours. As with Test 15 there was decontamination of the picrate species to a DRE of 99.999 percent or greater but unidentified compounds were still present. Characterization and identification of the compounds would require analytical efforts outside the scope of this project.

For Test 24 and 29 the control temperature remained at 600°F (316°C) but the temperature time was reduced again to 6 hours for the same conditions as Test 15. All of the projectiles were decontaminated to a DRE of 99.999 percent or greater.

Chamber Wipe Samples

Explosive residues were detected in only two of forty samples with these two residues located in the "rail" sample. These samples followed the trend from other tests of residues remaining on the rail due to rail acting as a heat sink and allowing cooler surfaces to be present. The average amount of residue was 2.3292E-05 mg/sq cm, which is in the same magnitude as the projectile extraction samples. Those samples indicated DREs of 99.999 percent or higher. It is a safe assumption that the chamber wipe samples

from Test 10, 15, 20, 24, and 29 indicate there is no accumulation of explosive residue within the chamber.

CEM Trends

As the projectiles approached 500°F (260°C) small peaks were noted on the CEM for CO and NO_x during all five tests. In Test 29 these peaks occurred during the time of temperature peaks when individual projectiles were going through thermal degradation. CEM and temperature peaks were likely occurring simultaneously in earlier tests, but was unobserved on the profiles due to the 15 minute sample intervals.

The usual peaks and drop-offs occur in Test 24 and 29 from a power interruption and a system shutdown caused by high collector temperature. Generally the broad bands in CO and NO_x levels correspond to temperature drops caused by the HGD system letting in outside air to compensate for temperature changes.

Once the system goes into steady-state operating the levels of CO, NO_x and total hydrocarbons return to usual background levels.

4.2.9 Test C

This test was conducted on an empty chamber to decontaminate the chamber of any explosive residues that may have accumulated during Test 1 through 10. This was a scheduled test as a precautionary measure to minimize the chance of deposition of explosive on chamber insulation. The need for such precaution is indicated by the construction of the chamber which has open seams in the interior walls, with the possibility of explosives penetrating into the chamber insulation. Test conditions were 24 hours at 700°F (371°C).

Chamber Wipe Samples

Explosive residues were detected in three of eight samples with an average quantity of 1.2681E-05 mg/sq cm. From the low quantities of explosives

residues detected in the chamber wipe samples it was decided that the chamber did not require decontamination as originally planned. These low quantities indicated that any explosives residues deposited on a surface at the conclusion of one test would re-volatilize as the next test came up to temperature. Therefore there was not a chance for an accumulation of explosives. The remaining scheduled chamber decontamination tests were eliminated from the demonstration program.

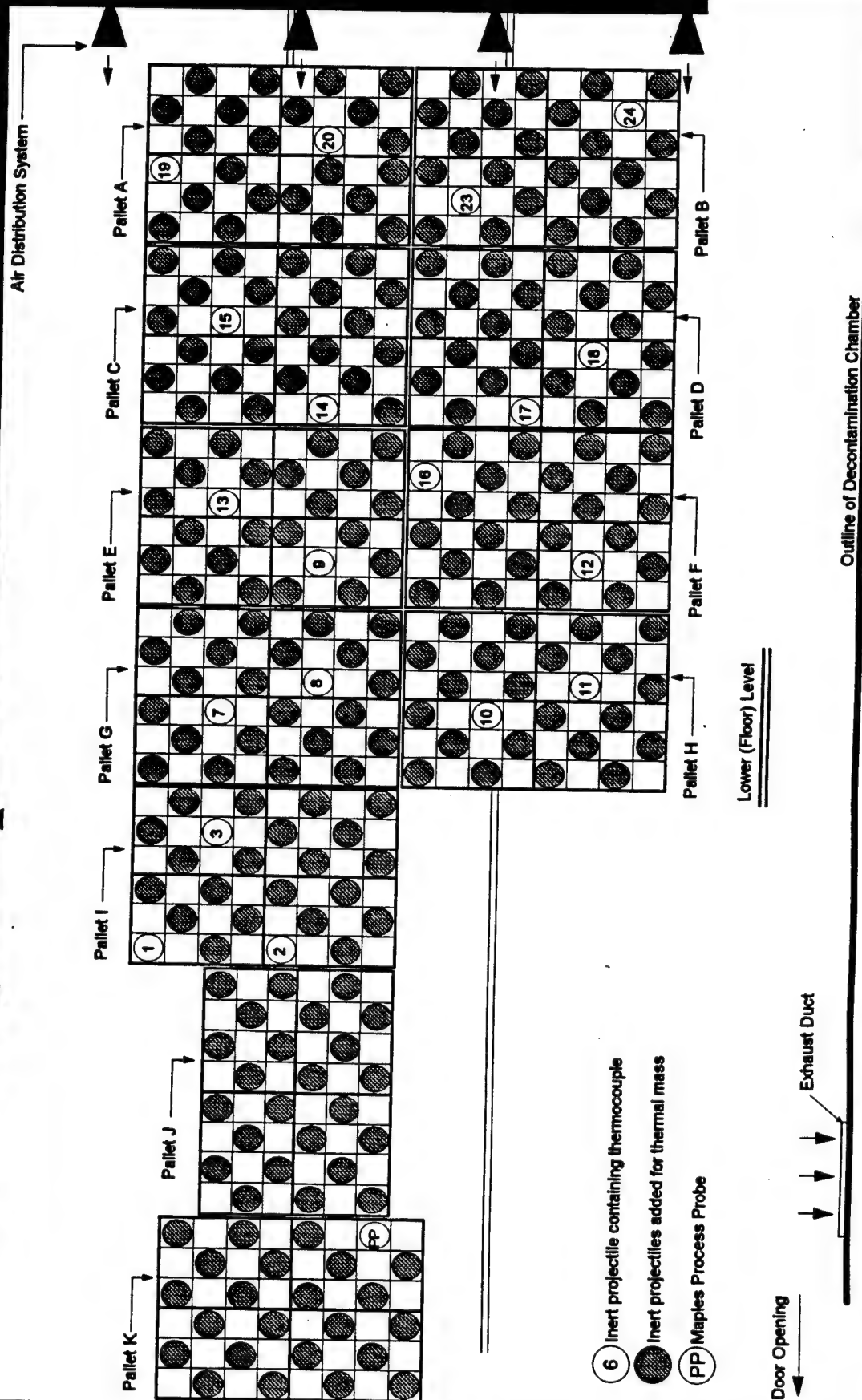
4.2.10 Test D

This test was conducted to establish the temperature distribution of 480 inert 175mm projectiles in a full chamber load configuration as shown in Figure 4-14 and 4-14a and Photo 4-13. Twenty-four projectiles were palletized on a standard 40 inch by 48 inch (102 centimeter by 122 centimeter) metal pallet by placing one projectile in every other position of a 5-inch projectile nose adapter pallet (Figure D-6a). The chamber accommodated 20 pallets in two tiers, two wide, extending lengthwise from the chamber exhaust duct to within 44 inches (112 centimeters) of the diffusers. The pallets were placed in the chamber with a forklift. Test conditions were 6 hours at 550°F (288°C).

The temperature data from this test was used to locate the temperature control element in the area in the projectiles which was the coldest. The temperature control element then remained fixed for all the full chamber load tests (Tests 30 - 34). Projectiles in other areas of the chamber were likely to be treated at a higher temperature than the nominal test temperature.

Average temperatures of the projectiles ranged from 505 to 800°F. Low temperatures were in the lower tier, center and back (towards the door); high temperatures were in the upper tier, front (near the diffusers). The coldest projectile was located at thermocouple #10 (Figure 4-14).

175mm Projectiles



See Figure D-6a for 5-inch adaptor placement on pallet

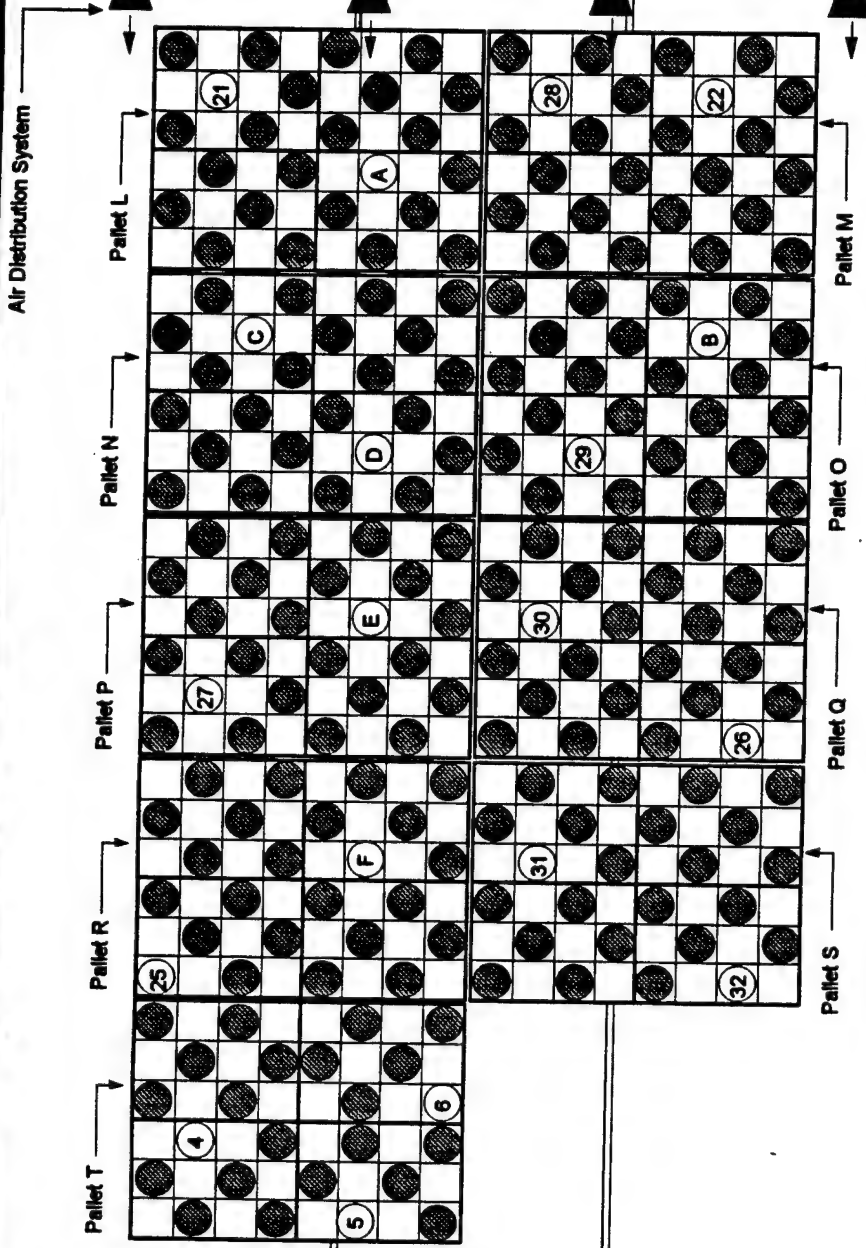
Test D
(Not to Scale)

8 Sep 1994
6 Hours at 550 °F / 288 °C

Figure 4-14 175mm Projectiles (Inert) - Lower Level - Full Chamber Load

175mm Projectiles (Items from FF-13)

- 6 Inert projectile containing thermocouple
- Inert projectiles added for thermal mass
- A D/L Point #2
- B D/L Point #3
- C D/L Point #4
- D D/L Point #5
- E D/L Point #6
- F D/L Point #7



Upper Level

Outline of Decontamination Chamber

Test D
(Not to Scale)

See Figure D-6a for 5-Inch adaptor placement on pallet

8 Sep 1994
6 Hours at 950 °F / 288 °C

Figure 4-14a 175mm Projectiles (Inert) - Upper Level - Full Chamber Load

Because inert projectiles were being used for this test projectile extraction samples and chamber wipe samples were not collected. For the same reason the CEM monitors were not in use.

4.2.11 Explosive: Comp A-3

General Observations

The final classification of explosive type tested during this demonstration program was Comp A-3. These residues were decontaminated in Test 22 and 28. The tests were performed with 106mm projectiles in racks placed on a railcar. Each railcar contained 168 inert projectiles for thermal mass and 24 melted out projectiles which were internally monitored for temperature. They were arranged on a railcar generally as shown in Figure 4-15 and in Photo 4.1. The location of the melted out projectiles in the racks was varied in the two tests to provide temperature monitoring and sampling over as many locations on the railcar as possible.

Projectile Extraction Samples

At a treatment time of 6 hours and a control temperature of 550°F (288°C), there was decontamination to a DRE of 99.999 percent or better. The average projectile temperatures for the 6 hours ranged from 578 to 650°F (303 to 343°C).

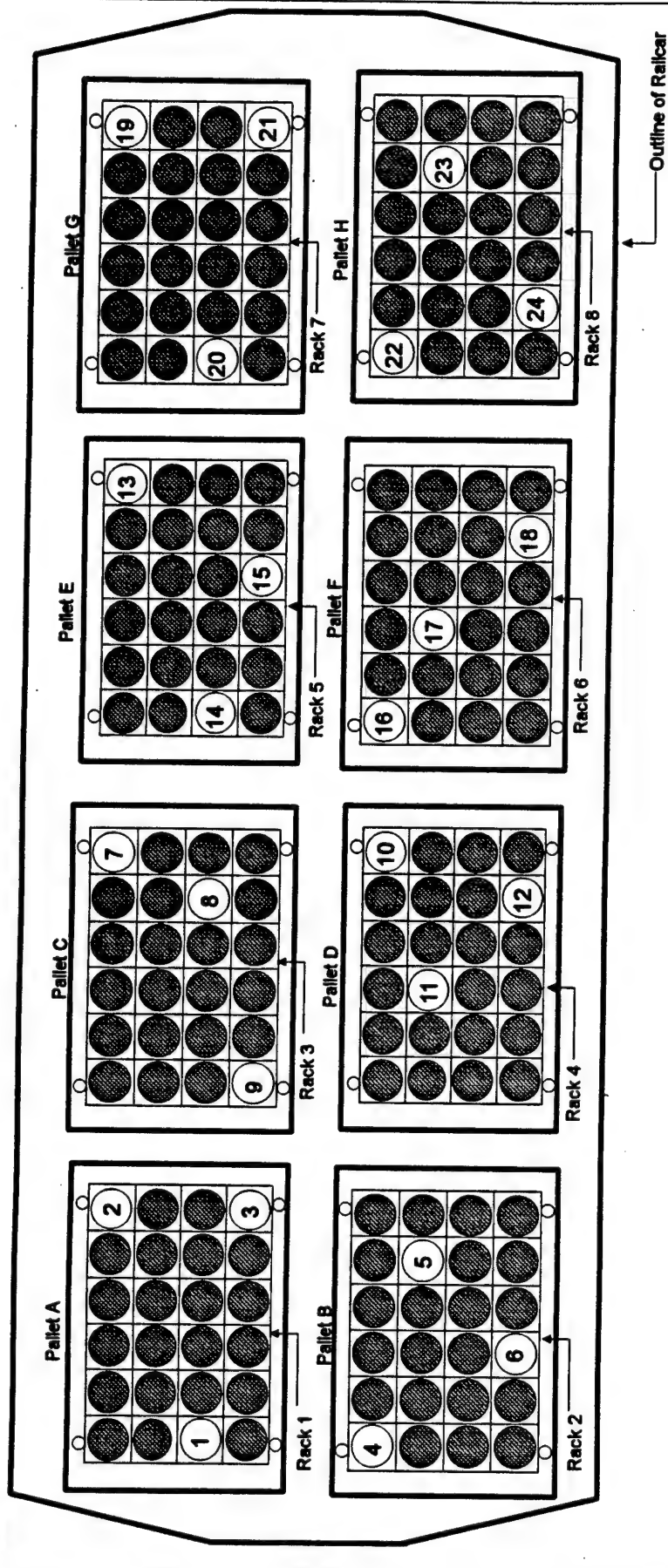
Chamber Wipe Samples

Explosive residues were detected in only one of sixteen samples and the quantity (1.6589E-05 mg/sq cm) was similar to the quantities detected in the projectile extraction samples. Based on these analyses it appears that Comp A-3 leaves very little explosive residue inside the chamber. These levels can be equated generally to the DRE stated above and that there is no accumulation of explosive residue within the chamber.

Total Weight, 192 projectiles = 10,560 lbs (4,790 kg)

106mm Projectiles (Projectiles contained Comp A-3 residue)

- 7 Contaminated projectile sampled
- Inert 106mm projectile added for thermal mass



See Figure D-1 for railcar placement in chamber
See Figure D-2 for pallet placement on railcar
See Figure D-5 for 5-inch rack placement on pallets

(Not to Scale)

Figure 4-15 106mm Projectiles with Comp A-3 Residue

CEM Trends

There were NO_x and CO peaks noted during heatup of the projectiles in Test 22. These increased levels decreased after the system went into steady-state operation and generally followed the baseline conditions. There were no obvious peaks after steady-state.

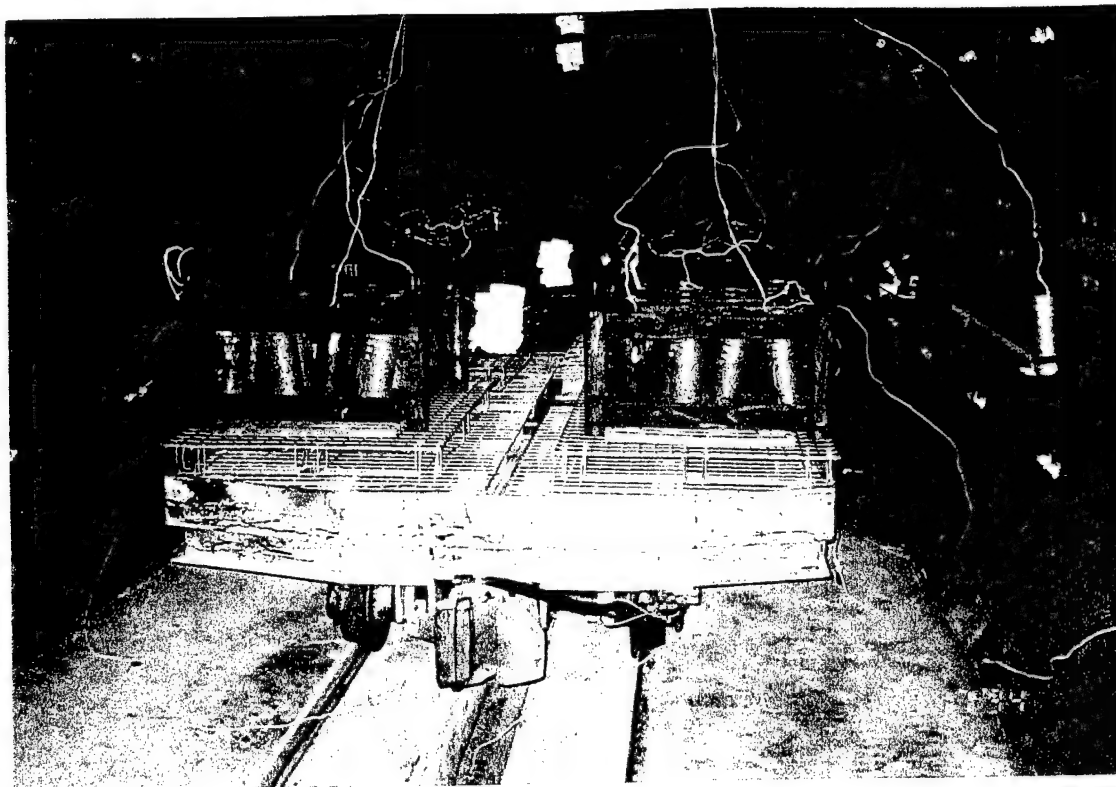


Photo 4-1: Railcar Configuration of 106mm Projectiles Containing Comp A-3 Explosive Residue (Tests 22 and 28).

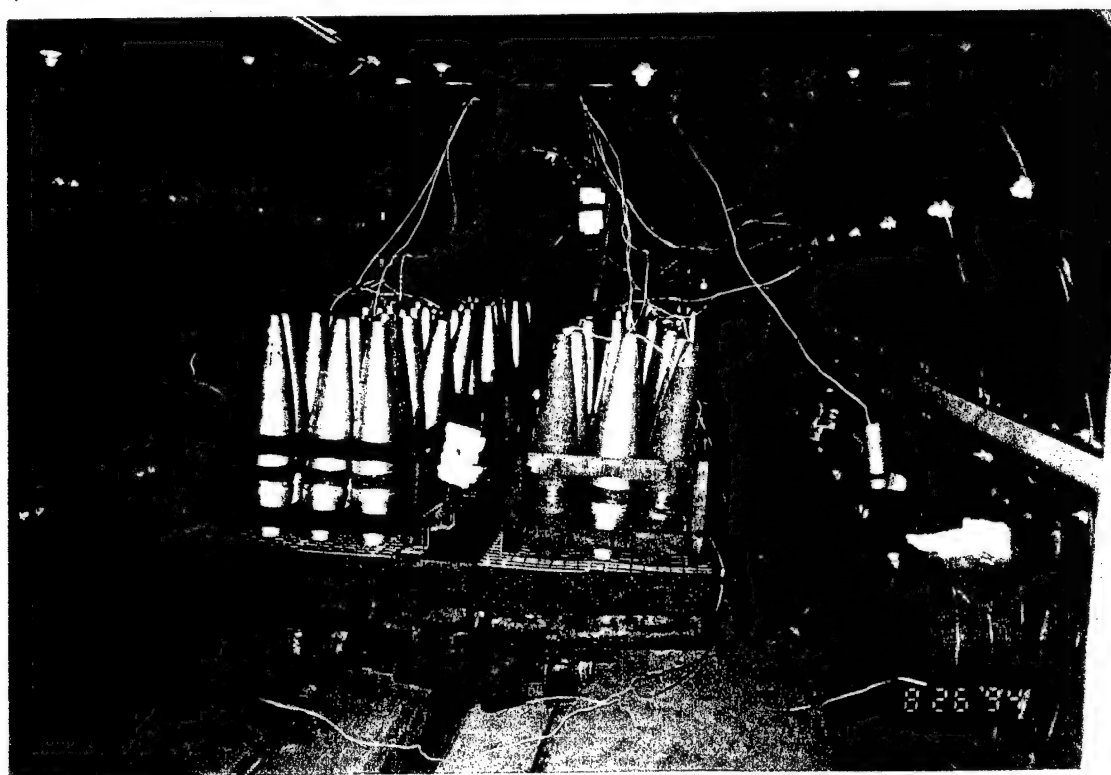


Photo 4-2: Railcar Configuration of 175mm Projectiles Spiked With Comp B Explosive (Tests 3, 8, 13, 18, and 23).

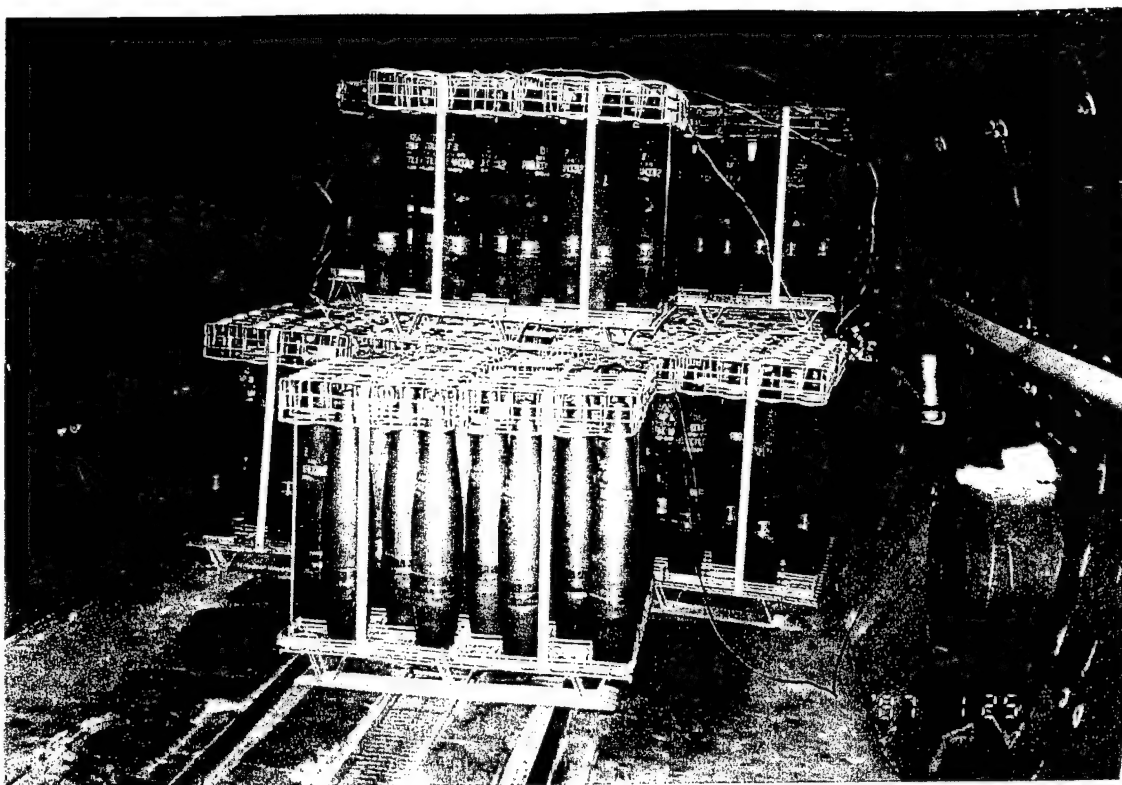


Photo 4-3: Full Chamber Load Configuration of 175mm Projectiles Containing Comp B Explosive Residue (Tests 30, 31, 32, 33, and 34).

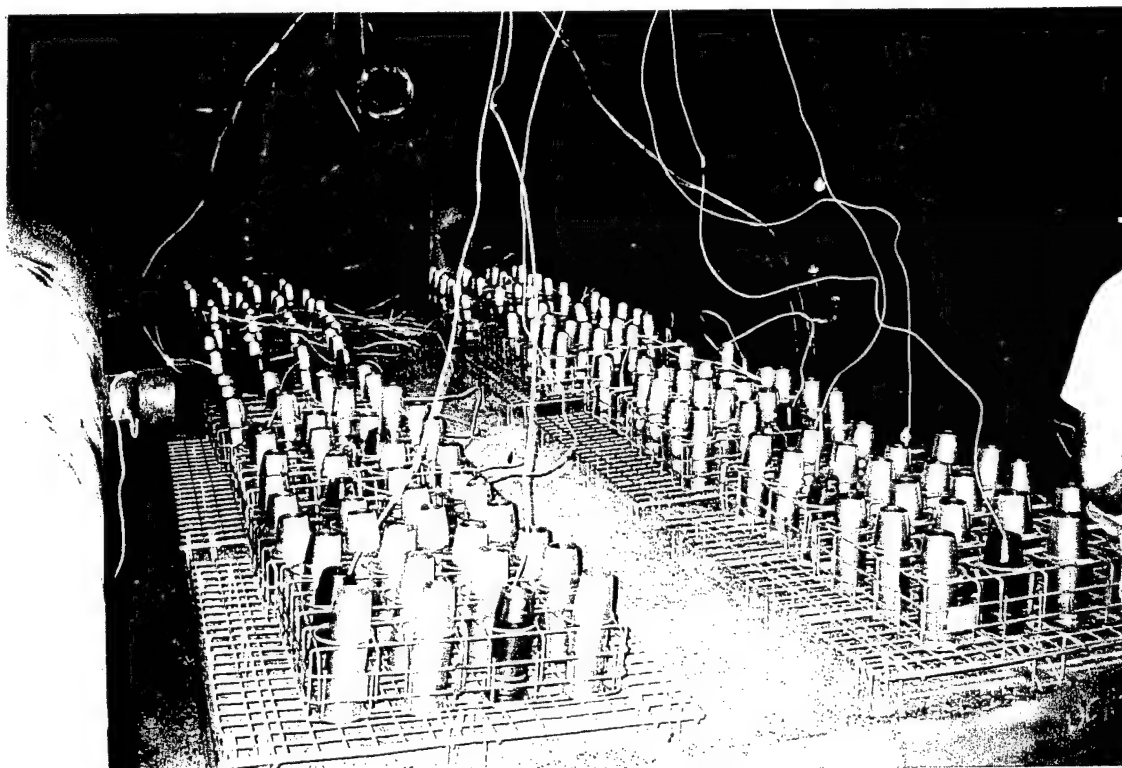


Photo 4-4: Railcar Configuration of 3-Inch Projectiles Spiked with HBX Explosive (Tests 5, 8, and 14).

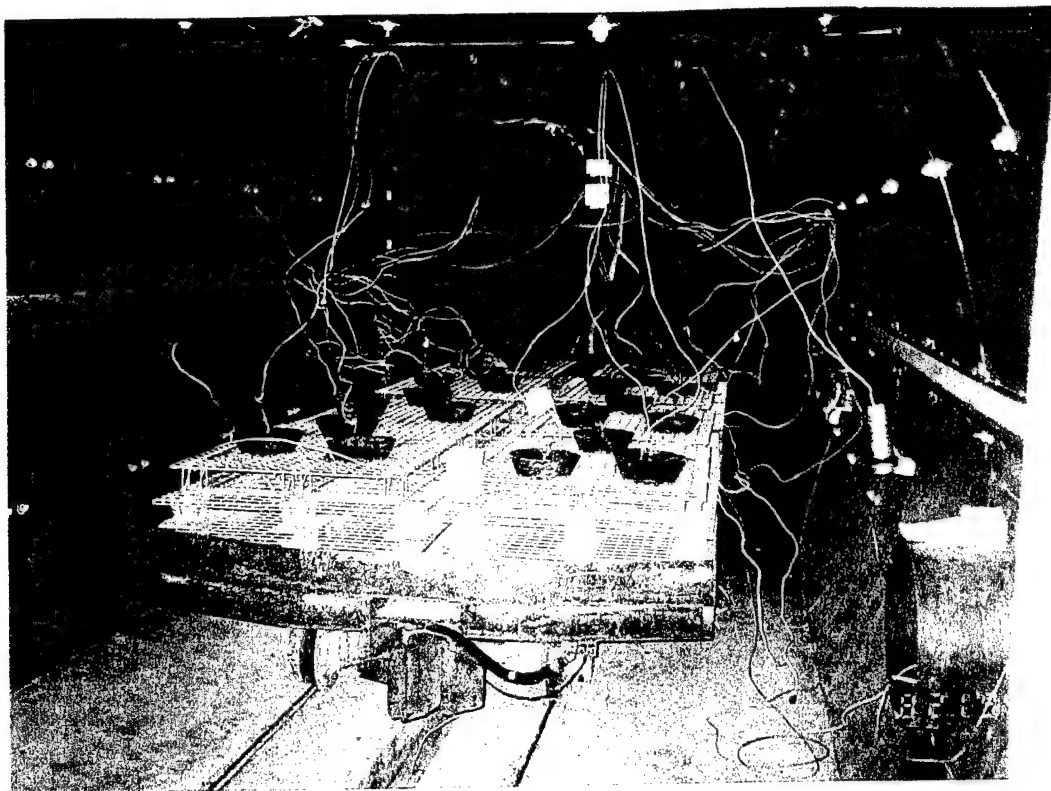


Photo 4-5: Railcar Configuration of MK 54 Depth Bombs (Sawed Ends) Containing HBX Explosive Residue (Tests 16, 19, 21, and 25).

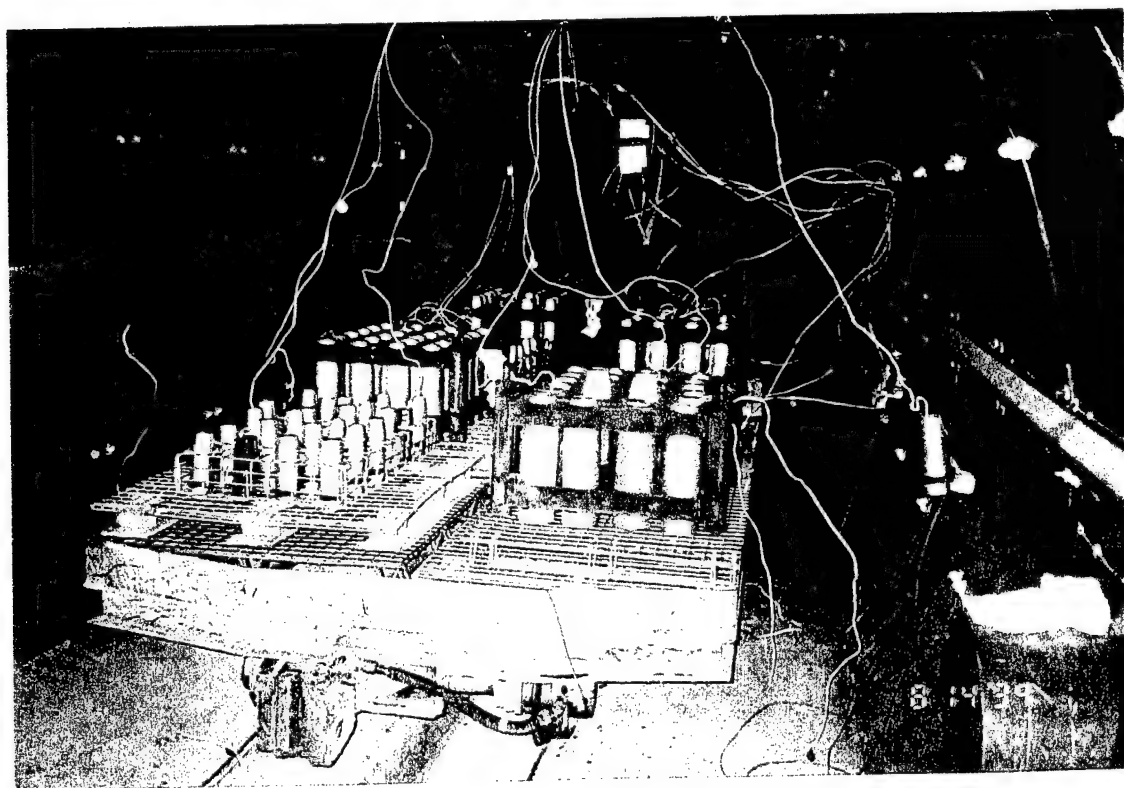


Photo 4-6: Railcar Configuration of 3-Inch and 5-Inch Spiked Projectiles Spiked With RDX Explosive (Tests 2, 7, 12, and 17).

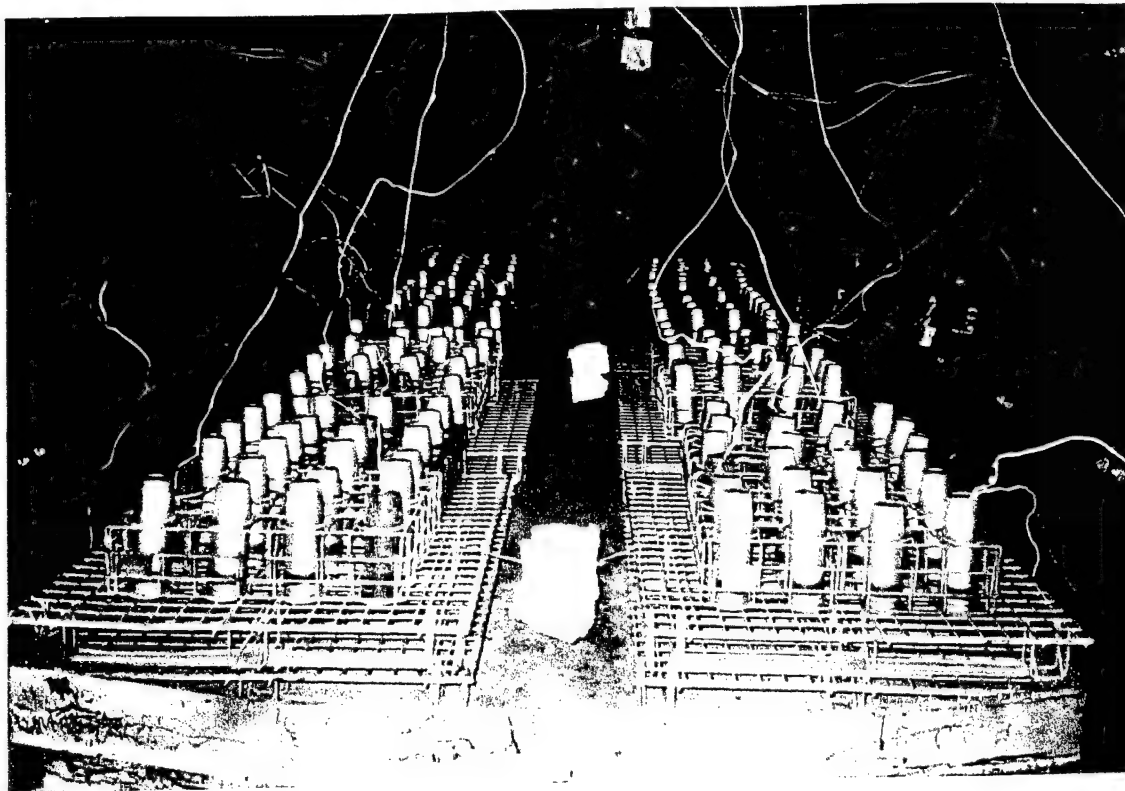


Photo 4-7: Railcar Configuration of 3-Inch Projectiles Spiked with TNT Explosive (Tests 1 and 4).

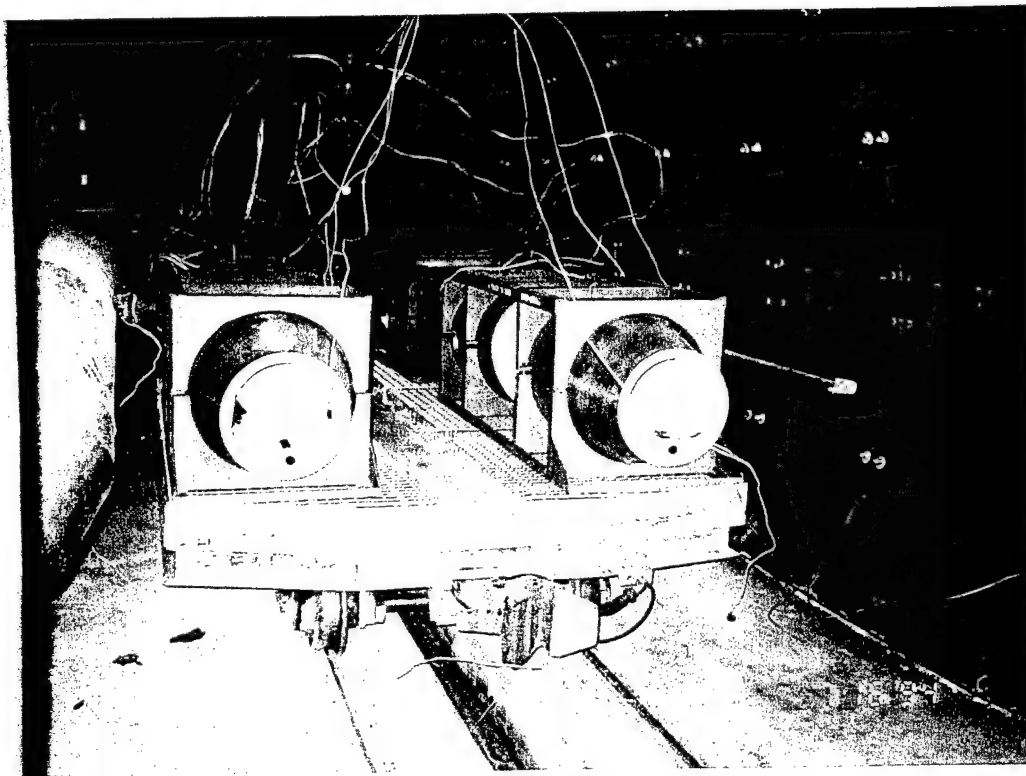


Photo 4-8: Railcar Configuration of MK 25 Ship Mines (Sawed in Half) Spiked with TNT Explosive by TVA (Tests 6 and 11).

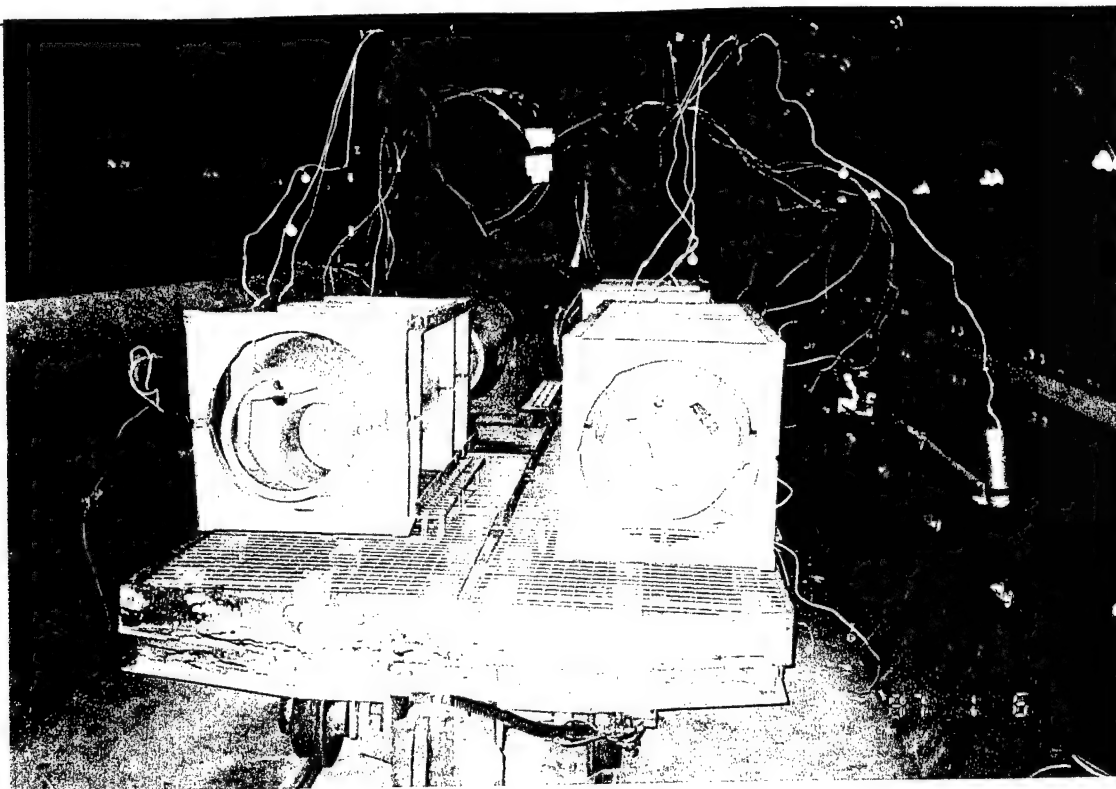


Photo 4-9: Railcar Configuration of MK 25 Ship Mines Spiked With TNT Explosive by Weston (Test 26).

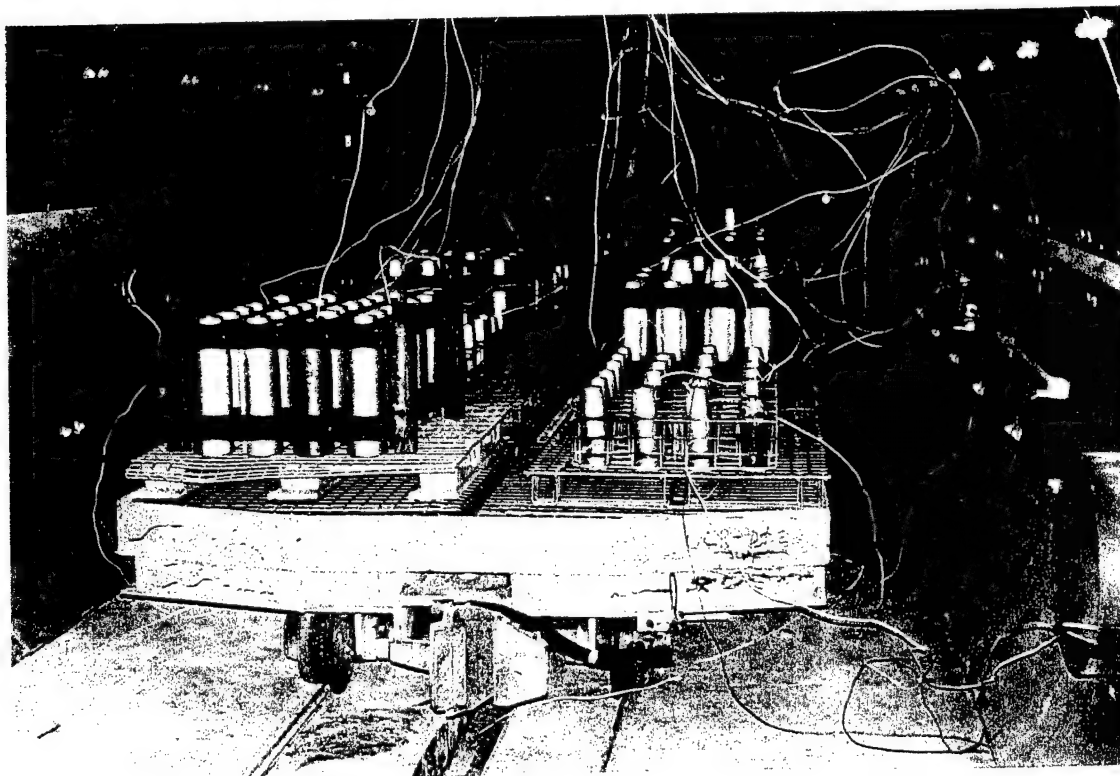


Photo 4-10: Railcar Configuration of 3-Inch and 5-Inch Projectiles Spiked with Yellow D Explosive (Tests 10, 15, 20, 24, and 29).

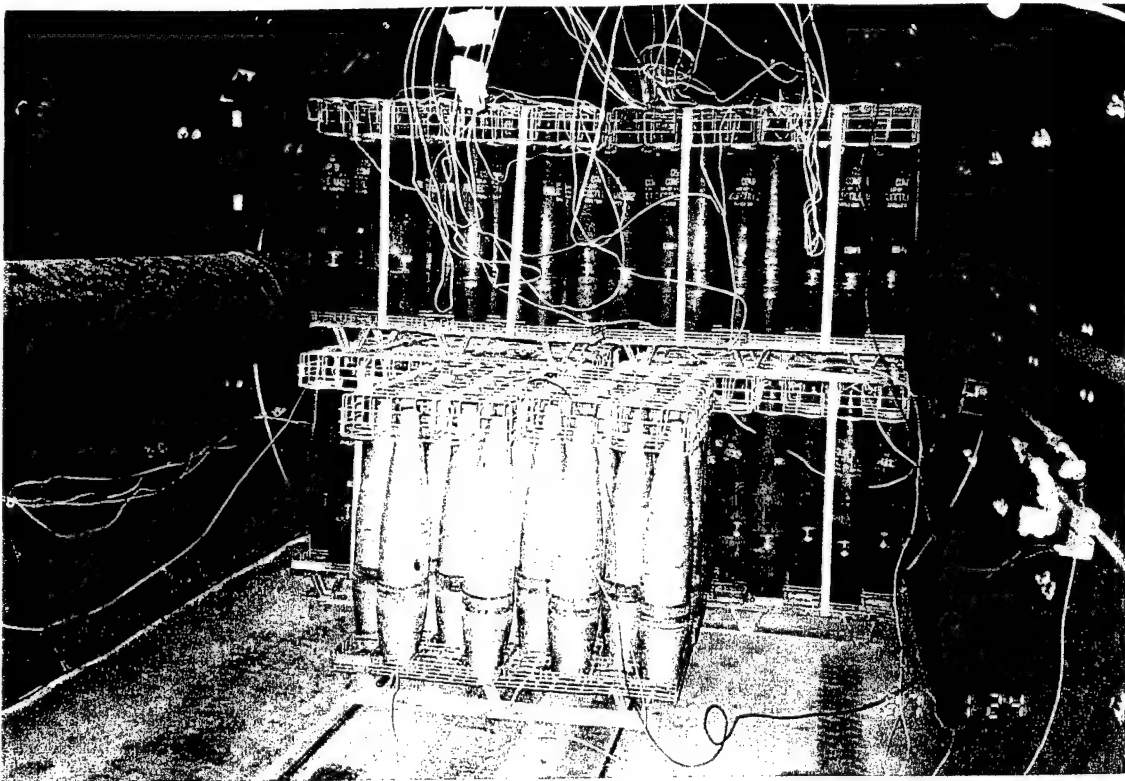


Photo 4-11: Partial Chamber Load Configuration of 175mm Projectiles Containing Comp B Explosive Residue (Test "96").

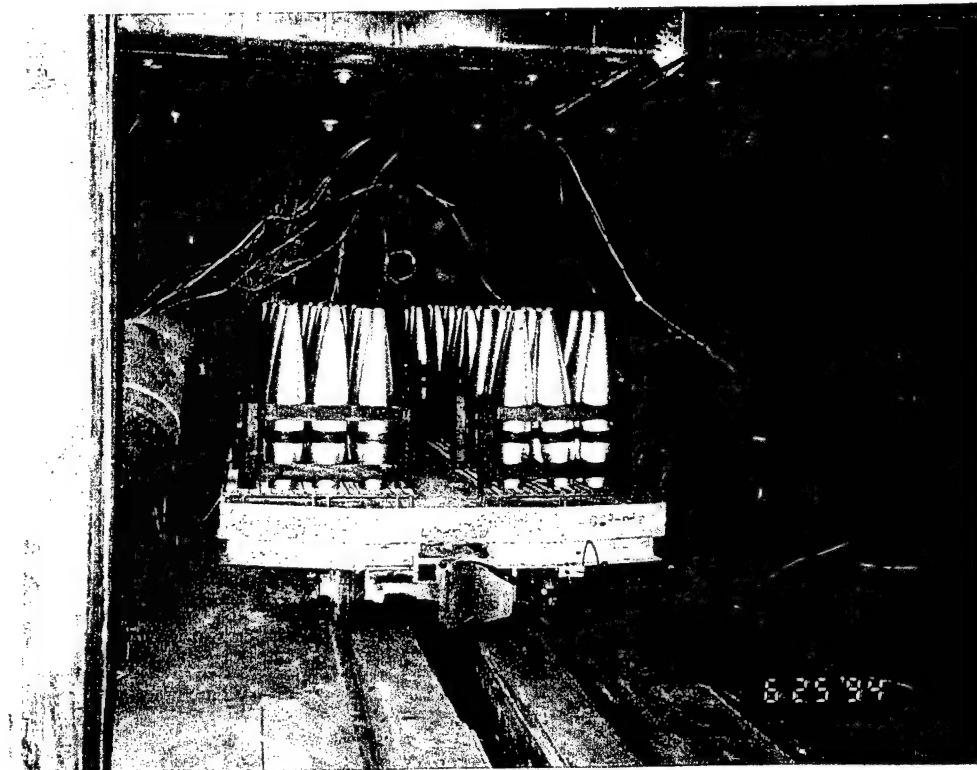


Photo 4-12: Railcar Configuration of Inert 175mm Projectiles (Test B).

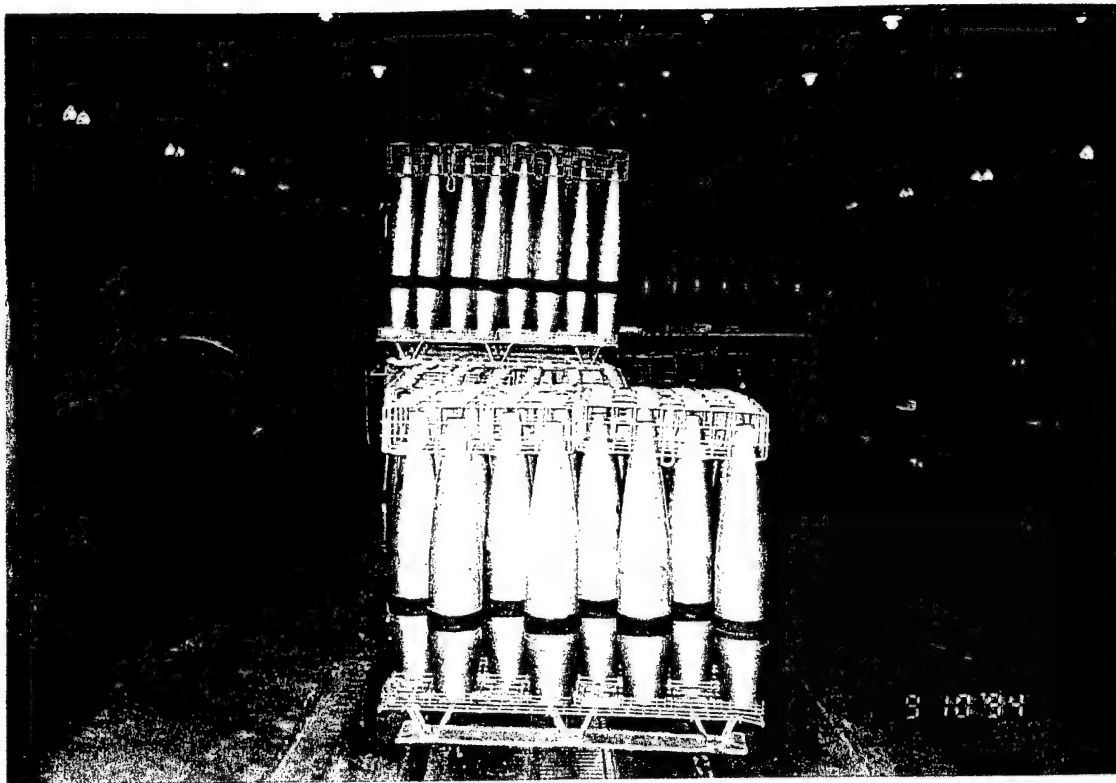


Photo 4-13: Alternative Configuration For Full Chamber Load of Inert 175mm Projectiles (Test D).

5.0 ECONOMICS

5.1 Introduction

These operating costs are based on data obtained from records at Hawthorne Army Depot and from data gathered during the demonstration period from June through October 1994. These figures will apply only to the hot gas decontamination facility at HWAD and are not intended to represent operating conditions or costs at other facilities. The final operating cost figures are expressed in dollars per ton (\$/ton) of metal decontaminated and have been calculated using information collected during the first five operational tests (Tests 30 through Test 34). These tests were full chamber loads of 175mm projectiles, 480 projectiles per test, contaminated with Comp B, and represented quantities expected during production runs. A production run of 480 projectiles, each weighing 115 pounds (52.2 kg), is equivalent to approximately 55,200 pounds (25,038 kg) or 27.6 short tons (25.06 metric tons). Figures D-49 through D-53 indicate arrangement pallets of projectiles inside the decontamination chamber.

5.2 Cost Data

The following cost items shown in Table 5-1 were used to calculate the overall operating cost for each unit weight of material decontaminated:

Table 5-1
Unit Cost Data

Cost Item		Unit Cost ⁽⁶⁾
Personnel Costs	Material Handler	\$44.94/hour ⁽¹⁾
	Maintenance Laborer	\$48.86/hour ⁽¹⁾
Consumable cost	Fuel	\$.6004/gallon ⁽²⁾
	Electricity	\$0.549/kWh ⁽³⁾
	Maintenance Material	\$75.00/week ⁽⁴⁾
	Miscellaneous	\$75.00/week ⁽⁴⁾
Sample/Analysis	2 Samples Per Week	\$80.00/Sample
Cycle Time	1 Chamber Load = 40.6 hours ⁽⁵⁾	
	3 Chamber Loads Per Week ⁽⁵⁾	

⁽¹⁾ Hourly rates include fringe benefits, overheads, and contractor's profit.

⁽²⁾ Cost per gallon of DF#2, delivered to job site.

⁽³⁾ Average kWh cost (from HWAD) for September 1994 through March 1995.

⁽⁴⁾ Assumed cost (allowance) for materials and supplies.

⁽⁵⁾ Based on reliability of HWAD system.

⁽⁶⁾ All costs are shown in FY 95 dollars.

5.3 General Assumptions

To calculate a unit cost for Hot Gas decontamination of 175mm projectiles, the assumptions as shown in Table 5-2 were made:

Table 5-2

Assumptions Used in Cost Calculations

Man Hours	Materials Handler - 40 hours per week with no overtime required. Maintenance - 2 man-hours per week.
Consumables	Fuel Oil - DF#2 usage for Test 30 through Test 34 averaged 1,235 gallons (4,675 liters). For 3 tests each week 3,705 gallons (14,025 liters) would be required.
	Electricity - Three fans running essentially full time for each full chamber load, system instrumentation, and miscellaneous facility lighting would require approximately 767 kWh per load or 2,300 kWh per week.
	Miscellaneous Materials - \$75.00 per week.
	Miscellaneous Supplies - \$15.00 per week.
	Sampling/Analysis - 3 samples per week at \$80.00 per sample.
	Cycle Time - 3 full chamber loads per week. See Section 6.2.1 (Recommendations) for suggestions on facility operation and timing sequence for production runs.
Projectiles Decontaminated (Weight)	Each 175mm projectile weighs 115 pounds (52.2 kg). Each decontaminated load contains 480 projectiles. Three loads produce 1,440 projectiles per week. Weekly output equals 82.8 short tons (75.18 metric tons).

5.4 Cost Calculations

Using the unit cost data from Table 5-1 and the assumptions listed in Table 5-2, the weekly operating costs were calculated and are shown in Table 5-3. These weekly costs are summarized in Table 5-4. The total weekly cost shown in the summary table (\$4,576.07) was divided by the weekly output of 82.8 short tons (75.2 metric tons) of metal (175mm projectiles) to arrive at the final unit cost of \$55.27 per short ton (\$60.87/metric ton).

Table 5-3
Weekly Operating Cost

Cost Item	Unit Cost*	Weekly Cost*
Personnel -		
Material Handler	\$44.94/hr	\$1,797.60
Maintenance Laborer	\$48.86/hr	\$97.72
Subtotal		\$1,895.32
Material -		
Fuel Oil	\$.6004/gal	\$2,224.48
Electricity	\$.0549/kWh	\$125.27
Maintenance Material	\$75.00/week	\$75.00
Miscellaneous Supplies	\$15.00/week	\$15.00
Subtotal		\$2,440.75

Table 5-4
Operating Cost Summary

Cost Item	Total Cost*
Personnel Cost	\$1,895.32
Material Cost	\$2,440.75
Sampling	\$240.00
Total	\$4,576.07 per week

*All costs are shown in FY 95 dollars.

Total cost per short ton of metal = \$55.27 (\$60.87/metric ton)

5.5 Conclusions

The estimated total cost for the HGD process is \$55.27 per short ton (\$60.87/metric ton) of metal decontaminated. A considerable savings will be realized by using the hot gas decontamination facility to remove Comp B from washed-out 175mm projectiles. Additional time and materials savings is possible if the Wash Out Facility (Building 117-5) will reload the 175mm projectiles on 5-inch metal adapters and properly based racks suitable for the hot gas chamber. This will eliminate multiple handling of the wooden pallets containing 6 projectiles. The 175mm projectiles will be allowed to remain on the metal racks and adapters until delivered to the scrap yard for sale. The metal pallets and adapters may then be returned

to the Wash-Out facility for re-use. Sufficient pallets and adapters are on hand or in stock at HWAD.

By using the decontamination facility for the heavy 175mm projectiles, the wear and tear on the flashing furnace, conveying system, and rail cars will be greatly reduced. Additional savings are possible by completing a system optimization study prior to full scale production with 175mm projectiles.

5.6 Summary

The projected cost per short ton of metal decontaminated is \$55.27 (\$60.87/metric ton). Additional cost savings are possible by completing an optimization study and adjusting current operating procedures at the Wash-Out facility (Building 117-5).

6.0 Conclusions and Recommendations

6.1 Conclusions

6.1.1 Hot Gas Decontamination Concept

The Hot Gas Decontamination (HGD) Demonstration Program at Hawthorne Army Depot demonstrated the efficacy, safety, and economics of the HGD process to remove and destroy residues of explosives from obsolete munitions as the final step of the demilitarization process. Optimization of the HGD process as to minimum treatment time and temperature combinations necessary to decontaminate munitions were not within the scope of this test program because of budget and time restraints.

- The HGD process effectively decontaminated munitions spiked with explosives to microgram quantities of explosive residues, with a destruction and removal efficiency (DRE) greater than 99.999 percent. The HGD process also effectively decontaminated melted out and hot melt coated munitions to microgram quantities of explosive residues similar to that achieved with spiked munitions.
- There was no accumulation of explosive residues in the system during this demonstration program that could present a hazard to personnel or the facility. Only random microgram quantities of explosive residues were detected in wipe samples taken after each test, accumulative wipe samples taken after a series of ten tests, and an accumulative wipe sample taken after the last test.
- There was no accumulation of explosive residues on insulation test samples placed within the chamber. Only random microgram quantities of explosive residues were detected in accumulative block samples taken after every four to seven tests. Therefore, future HGD facilities will not require a metal

liner within the chamber but can utilize exposed insulation as currently used in contaminated waste processors.

- Emission rates of total hydrocarbons, SO₂, NO_x, CO, particulates, and metals from the HGD system during Comp B explosive production mode operation were significantly below state of Nevada allowable limits.

CEM test data indicates the feasibility for continuous monitoring of the HGD process effluent to determine when decontamination is complete. Increased NO_x and CO activity was well defined for explosives Comp B, HBX, and Ammonium Picrate. This activity was not as well defined for Comp A-3 and TNT explosives. Increased CO activity was observed during volatilization of the hot melt when testing MK 25 Ship Mines and MK 54 Depth Bombs. CEM data from Comp B explosive testing showed increased NO_x and CO activity during the time period when the projectiles neared 400°F/204°C. NO_x and CO activity returned to baseline levels after all the projectile temperatures surpassed 400°F/204°C. This would indicate decontamination was complete. Tests and sample analysis to confirm decontamination were not within the scope of this test program.

Temperature and CEM spikes indicate that the mechanism of decontamination for all of the explosives tested is more complicated than volatilization and most likely thermal degradation.

Individual projectile temperatures were compared to the quantity of explosive residue remaining in that projectile after treatment. It was thought that projectiles with higher treatment temperatures would exhibit less explosive residue after treatment. However, test data did not show a correlation between increasing projectile temperatures and a decrease in explosive residue concentration remaining after treatment.

6.1.2 Hawthorne Facility

In prior testing Weston (Reference #2 RFW Report) demonstrated that the HGD process would decontaminate residues of TNT and ammonium picrate from various items singly or in small lots. The present tests extended the process to additional munitions and explosives and tested them in quantities approaching the projected usage of the facility in routine operations.

- The test program was comprised of 34 tests of five explosives (Ammonium Picrate, Comp A-3, Comp B, HBX, and TNT) and six munitions (3-inch, 5-inch, 106mm, and 175mm projectiles, MK 25 ship mines, and MK 54 depth bombs) with treatment times of 6 to 32 hours and treatment temperatures of 550 to 700°F/288 to 371°C. The HGD process effectively decontaminated these munitions to microgram quantities of explosive residues, with a destruction and removal efficiency (DRE) greater than 99.999 percent. The operational parameters at steady state conditions for each explosive are as follows;

Table 6.1
Operational Parameters at Steady State Conditions

Comp A-3	6 hours at 550°F/288°C
Comp B	6 hours at 550°F/288°C
HBX only	6 hours at 600°F/316°C
HBX over hot-melt ^(a)	24 hours at 700°F/371°C
RDX	6 hours at 550°F/288°C
TNT only	6 hours at 550°F/288°C
TNT over hot-melt ^(a)	32 hours at 700°F/371°C ^(b)
Ammonium Picrate	6 hours at 600°F/316°C

^(a)Reference Section 3.5.2.

^(b)Reference Section 4.2.2.2.

- There was no accumulation of explosive residues within the facility (chamber, fan, ductwork, etc.) during this demonstration program that could present a hazard to personnel or the facility.
- Emission rates of total hydrocarbons, SO₂, NO_x, CO, particulates, and metals from the HGD system during full load chamber tests of melted-out 175mm projectiles containing Comp B explosive were significantly below state of Nevada allowable limits. The estimated quantity of Comp B explosive decontaminated during each test was 9600 grams. The SO₂ emissions were produced by the diesel fuel used to fire the thermal oxidizer.

The operational procedure of day shift handling of munitions, with automated, unattended overnight heatup, steady state treatment, and cooldown of the chamber worked well. The process control system and safety interlocks functioned properly. Adjustments to the Maples controller during the early tests in the test sequence improved steady state operation with fewer drop-outs from the control temperature setpoint. The addition of a power surge protector could prevent interruptions in operation due to power surges.

- The total O&M costs for HWAD's HGD facility compare very favorably to the O&M costs for operating the flashing furnace.

6.2 Recommendations

6.2.1 HWAD Facility Operation

The operational procedure of day shift handling of munitions, with automated, unattended overnight heatup, steady state treatment, and cooldown of the chamber worked well.

For full chamber load decontamination of 175mm projectiles containing Comp B explosive, the heatup time was 18 hours, the process time was 6 hours, and the cooldown time to a chamber temperature of 150°F/66°C was 18 hours. With chamber unloading and loading, two days would be needed to process a batch. A recommended full chamber load operational procedure would be;

Table 6.2
Recommended Operational Procedures
Full Chamber Load

	<u>Time</u>	<u>Activity</u>
Monday	0800 - 1100 hours	Unload chamber
	1100 - 1400 hours	Load chamber
	1400 - 0800 hours	Heatup
Tuesday	0800 - 1400 hours	Steady State
	1400 - 0800 hours	Cooldown
Wednesday	0800 - 1100 hours	Unload chamber
	1100 - 1400 hours	Load chamber
	1400 - 0800 hours	Heatup
Thursday	0800 - 1400 hours	Steady State
	1400 - 0800 hours	Cooldown
Friday	0800 - 1100 hours	Unload Chamber
	1100 - 1400 hours	Load Chamber
	1400 - 0800 hours	Heatup
Saturday	0800 - 1400 hours	Steady State
	1400 - 0800 hours	Cooldown

Heatup, steady state, and cooldown phases on Saturday would be unattended operation.

For railcar decontamination of munitions, heatup times ranged from 4 to 20 hours, process times ranged from 6 to 32 hours and cooldown times ranged

from 8 to 14 hours. General guidelines for railcar load operational procedures follow:

Table 6.3
Operational Procedure Guidelines
Railcar Load

<u>Munition</u>	<u>Explosive</u>	<u>Time (in hours)</u>		
		<u>Heatup</u>	<u>Process</u>	<u>Cooldown</u>
175mm	Comp B	7	6	12
3/5 inch	Yellow D	8	6	10
106mm	Comp A-3	4	6	8
MK 25 Ship Mine	TNT	20	32 ^(a)	12
MK 54 Depth Bomb	HBX	12	24	14

^(a) Reference Section 4.2.2.2.

6.2.2 Control System

The addition of a power surge protector could prevent interruptions in operation due to power surges.

6.2.3 CEM

It is recommended in future HGD facilities that a CEM be installed to monitor the CO, NO_x, and THC process effluents from the decontamination chamber to determine when explosive decontamination is complete. The CEM could be used as a process control instrument to start the chamber cooldown after completion of the decontamination cycle.

However, for the HWAD HGD Facility, the installation of a CEM is not justified. Exhaust gases containing NO_x and CO effluents are circulated from the thermal oxidizer to the flash chamber and combined with effluents from thermal degradation of the explosives. It would be difficult to use the CEM to determine decontamination completion because of the variable levels of NO_x and CO effluents from the recirculated exhaust gases.

7.0 References

1. Final Report on "A Study of Equipment, Processes, and Systems for a Demilitarization Facility at NAD, Hawthorne, Nevada." Phase II, Vols. I, II, III, and IV by Battelle Columbus Laboratories, 1978.
2. "Task Order - 2 Pilot Test of Hot Gas Decontamination of Explosives - Contaminated Equipment at Hawthorne Army Ammunition Plant (HWAAP), Hawthorne, Nevada"; Report No. CETHA-TE-CR-900(?), July 1990, by Roy F. Weston Inc.
3. Final Report, "Hot Gas Decontamination Facility Shakedown Test, Hawthorne Army Ammunition Plant" by Day & Zimmerman/Basil Corporation, 31 October 1993.
4. Pilot Plant Testing of Caustic Spray Hot Gas Building Decontamination Process, Task Order #5, Final Report dated August 1987. Report # AMXTH-TE-CR-8712, Arthur D. Little, Inc., Authors: L. L. Woodland and A. A. Balasco.
5. TVA's "Hot Gas Decontamination of Explosives Contaminated Items, Process and Facility Conceptual Design, AEC Report No. SFIM-AEC-ET-CR-94118, dated January 1995.